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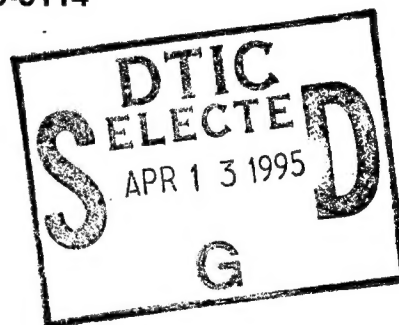


**PRIVATE WELL WATER SAMPLING PLAN TO MEET THE  
REQUIREMENTS OF ATSDRs PUBLIC HEALTH ASSESSMENT  
AT THE TUCSON INTERNATIONAL AIRPORT SUPERFUND SITE (TIASS)**

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**SEPTEMBER 1994**



**Final Technical Report for Period 21 - 24 May 1994**

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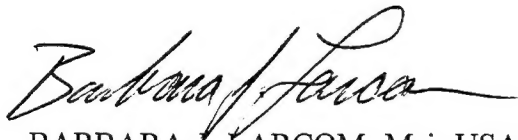
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REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE 1 Sep 94	3. REPORT TYPE AND DATES COVERED Final 21-24 May 1994		
4. TITLE AND SUBTITLE Private Well Water Sampling Plan to Meet the Requirements of ATSDRs Public Health Assessment at the Tucson International Airport Superfund Site (TIASS)			5. FUNDING NUMBERS	
6. AUTHOR(S) Guy Cornell Long Wade H. Weisman, Capt, USAF, BSC				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Armstrong Laboratory (AFMC) Occupational and Environmental Health Directorate Occupational Medicine Division 2402 E Drive Brooks Air Force Base, TX 78235-5114			8. PERFORMING ORGANIZATION REPORT NUMBER  AL/OE-TR-1994-0126	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)			10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION / AVAILABILITY STATEMENT  Approved for public release; distribution is unlimited			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words)  Armstrong Laboratory's Environmental Sciences Branch of the Occupational Medicine Division (AL/OEMH) helps Major Air Commands (MAJCOMs) and installations in preparing for public health assessments (PHAs). The Agency for Toxic Substances and Disease Registry (ATSDR) conducts PHAs at all National Priorities List (NPL) sites. Part of the preparation is to identify data gaps that may need to be filled for completion of a PHA. This sampling plan describes a sampling event in the South Tucson area; the area surrounds the Tucson International Airport Superfund Site (TIASS), the site of Air Force Plant 44. The plan outlines the approach used by AL/OEMH and Region IX USEPA and ATSDR to collect private well water samples. ATSDR will use the analytical data to assess exposure pathways for the PHA at the TIASS.				
14. SUBJECT TERMS ATSDR Private well water sampling Public health assessment			15. NUMBER OF PAGES 84	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL	

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## PREFACE

### Purpose

The purpose of the private well water sampling effort is to provide accurate exposure information to the health assessor from the Agency for Toxic Substances and Disease Registry (ATSDR) for the completion of the Public Health Assessment (PHA) at the Tucson International Airport Superfund Site (TIASS). The information from the survey will characterize the quality of drinking water of homes in the area of concern. This will be accomplished by specifically analyzing home tap water samples for the presence of trichloroethylene (TCE) and chromium (Cr) contamination. The area of concern for the survey is broadly described as an approximately one mile wide and three mile long corridor that is situated to the north and west of the TIASS. The area encompasses the known groundwater contamination associated with the TIASS site.

### Professional Responsibilities

The ATSDR TIASS Representative and the US Environmental Protection Agency (US EPA) Region IX Representative are responsible for determining the homes where sampling is required. The Pima County Department of Environmental Quality (PDEQ) Representative for this project will collect information about the well location, construction and usage, and schedule a window of time for the actual sampling. The Occupational Medicine Division of the US Air Force Armstrong Laboratory (AL/OEM) will prepare the workplan and submit it for approval through ATSDR, IX EPA, and PDEQ. Additional parties are included on the workplan review and approval sheet, page 6. Signatures on the plan indicate concurrence with the methods and procedures outlined in the plan. AL/OEM will collect the drinking water samples, provide the analysis, and ensure transmission of the analytical results directly from the analytical laboratory to ATSDR and EPA. As part of the actual field sampling (in addition to the collection of samples), AL/OEM will perform requested field measurements and ensure adequate chain of custody and quality assurance for the sampling efforts. The Pima County Department of Environmental Quality (PDEQ) will be responsible for the analysis and result reporting of the sample results from the split sampling accomplished. A representative from PDEQ will accompany the sampling team to ensure proper handling of the samples after they have been collected by AL/OEM. Upon conclusion of the sampling and analysis events, EPA and ATSDR will make conclusions and recommendations for further action.

### Acknowledgements

We would especially like to thank Pima County Department of Environmental Quality for helping us develop this plan and the many individuals who spent a great deal of time reviewing and commenting on the initial plan. This workplan was developed using the following documents as guides:

1. ABB Environmental Services, Inc., Residential Well Sampling and Analysis Plan, Loring AFB, Maine, March 1992.

2. The Air Force Center for Environmental Excellence (AFCEE) Workplan Guidebook.
3. Analytical Services Division, Armstrong Laboratory. Quality Management Guide, May 1990, p 113.
4. Armstrong Laboratory Sampling Guide, March 1989.
5. HAZWRAP Support Contractor, Quality Control Requirements for Field Methods, 6 July 1990.
6. Oak Ridge National Laboratory, Health and Safety Research Division, Field Sampling Methodologies Manual, 15 April 1993.
7. Pima County Department of Environmental Quality (PDEQ), November 1992 Private Well Sampling Plan.
8. BC Laboratories, Inc. CLP Project Manual, 5 Nov 93.

#### Period of Work

Field sampling will be performed after the PDEQ conducted door-to-door (DTD) well water use survey. AL/OEM, ATSDR, and EPA will agree on a time for the field sampling effort so occupants where samples are collected know when to expect the field sampling team. The length of the field sampling effort will be determined among the signatories of the plan. The tentative schedule for completing the survey is Apr 1994 and May 1994 for the sample collection.

## SAMPLING AND ANALYSIS PLAN - OVERVIEW

### Purpose

The purpose of the Sampling and Analysis Plan (SAP) is to describe the procedures used to sample well water from private taps in the area of concern near the TIASS especially those that have not been sampled within the past 12 months. In addition, the SAP provides the quality assurance parameters necessary to define the level of reliability of data generated to ensure the results can be used to complete the PHA. The main concern for this study is the protection of public health. As such, additional information about well water consumption rates, number of persons in the household, and length of time the people have lived at the home will be collected from residents to establish a potential exposure duration. This information will be collected during the DTD survey, which is considered separately from this workplan.

### Scope

Many of the wells in the area of concern have been tested prior to this effort. The importance of this field work is to sample every well in the area of concern near the TIASS. This area is indicated in Fig 1. The subjective criteria used to determine which wells to include in the survey were:

- 1) Proximity to the TCE plume
- 2) Results of past sampling efforts
- 3) Proximity to well (monitoring, private, or public) with known contamination.

These criteria were used to determine the wells that would need sampling. All wells located in the area of concern will be sampled during this field effort, even if they have been sampled previously. The wells to be included in this sampling effort include the priority 1-4 wells defined in the Nov 1992 PDEQ Private Well Sampling Plan, and also wells only used for irrigation or livestock watering. These categories of wells are described below (reproduced verbatim from the 1992 PDEQ plan):

Priority 1: Any well actively in use for human consumption, bathing, cooking, or clothes laundering, which (a) has not previously been identified or tested for contamination by any party involved in the investigation of the study area, and (b) lies within the zone of known contamination exceeding safe drinking water standards. This category includes wells used for irrigation only if the water is also used for human consumption, bathing, cooking, or clothes laundering.

Priority 2: Any well actively in use for human consumption, bathing, cooking, or clothes laundering, which (a) has previously been identified or tested for contamination but which has not been tested recently (*e.g.* within the past two years), and (b) lies within the zone of known contamination exceeding safe drinking water standards. This category also includes wells used for irrigation only if the water is also used for human consumption, bathing, cooking, or clothes

laundrying.

Priority 3: Any well actively in use for human consumption, bathing, cooking, or clothes laundrying, which (a) may or may not previously have been identified or tested for contamination and (b) lies within the general area of concern but outside the zone of known contamination. This category again includes wells used for irrigation only if the water is also used for human consumption, bathing, cooking, or clothes laundrying.

Priority 4: Any well actively in use for human consumption, bathing, cooking, or clothes laundrying, which has been tested recently or regularly for contamination.

The preliminary list of individual residences that have been identified for potential inclusion in the sampling efforts is being determined and will be available for the sampling team prior to starting work. Confirmation of the private well and its current operational status is necessary to include the residence in the well sampling program. As with the PDEQ plan, parties who receive their water supply only from Tucson water and do not own a private well (even if they are in the impacted area) will not have their water sampled during the field effort.

An additional qualification for the wells sampled is serviceability and present use. Wells that fall within the area of concern but are non-functional (i.e. sanded or silted in) will not be sampled. Wells that are functional, but are currently not being used for drinking water and other household purposes (i.e. the home is also connected to Tucson water) will be sampled as close as possible to the point of potential human contact. This type of well will require longer flushing prior to sample collection.

Inclusion of additional wells that fall outside the zone of concern, but in homes where the occupants have requested sampling, will be handled on a case-by-case basis. The inclusion of these wells will be discussed with ATSDR, EPA, and AL prior to adding them to this sampling effort.

## OBJECTIVES FOR MEASUREMENT CRITERIA

### Sample Analyte List

All samples collected will be analyzed for volatile organic compounds (VOCs) using EPA methodology 524.2. This method will indicate whether the sample contains TCE and other VOCs. Method 524.2 was selected to eliminate the need for a second column confirmation, as is needed for 502.2. Method 524.2 is a GC/MS method that provides confirmation in the form of a mass spectrometer finger print. In addition to the mass spectrometer confirmation, it is preferred by the lab performing the analyses for this project, BC Laboratories, because it lends itself better to Contract Laboratory Program (CLP) reporting. All samples will also be analyzed for total chromium using EPA method 218.2. Method 218.2 was selected to achieve practical quantitation levels (PQL) of 10 parts per billion, which is 1/10 of the EPA's maximum contaminant level (MCL). If the results for total chromium are 100 parts per billion (ppb) or greater, a follow-up sample will be collected and analyzed to speciate the chromium, using EPA

method 7196 and employing the same QA/QC and data validation as for total chromium. The speciation would indicate the percent of chromium VI in the total amount of chromium measured. Based on past drinking water samples collected from private homes in the area of concern, it appears that chromium contamination is not widespread. The 100 ppb threshold was selected since it is the total chromium MCL. In addition to the laboratory analysis of the water samples, field measurements will also be taken. The field measurements to be recorded at each well include: pH, temperature and conductivity. The primary purpose for the field measurements is to ensure proper purging of the water system. These measurements may also help to characterize the aquifer in which the well is screened since it is assumed that many of the residents will not know the depth of the screened interval for their well. The field measurements will be taken at every sampled residence.

### Quality Assurance/Quality Control

#### Program Data Quality Objectives

The level of quality control (QC) necessary for this field work is dependent upon the data quality objectives (DQOs) that define the level of reliability of data generated. Since the intended use of the information gathered during the sampling effort is to complete the PHA for the TIASS, the DQOs selected are intended to provide the quality of data needed for the PHA (i.e., the level necessary for completing risk assessments). The overall completeness goals for wells to be surveyed is 100%. This survey is not intended to compare past and current analytical information, but rather will serve as a snapshot of current potential contamination in the area. The precision goals for field duplicate analyses are 30% for 524.2 and 20% for 218.2. The laboratory selected for the chemical analysis of the drinking water samples is BC Laboratories of Bakersfield, CA. The accuracy goals for the project are documented in BC Laboratories Standard Operating Procedures (SOP) and Quality Goals, both contained in Appendix E. BC Laboratory will provide all documentation identified in the EPA Region IX, QAMS, guidance document, "Laboratory Documentation Requirements for Data Validation, DC# 9Qa-07-89, January, 1990". Validation of the sample results will be carried out by the US EPA, QAMS, following the "National Functional Guidelines for Inorganic and Organic Data Review".

#### Quality Control

BC Laboratory's QA/QC requirements are cited in the SOPs provided in Appendix E for methods 524.2 and 218.2. BC Labs will provide full QC documentation for all analysis directly to AL/OEM, Region IX EPA, and ATSDR.

Field Quality Control Samples. The level of field quality control was determined by the Region IX EPA Remedial Project Manager (RPM). These requirements are incorporated into this section. There are several types of QC samples including: trip blanks, equipment rinsates, field blanks, field duplicates, and split samples. Since the sample collection equipment used for the field work is single use and will not require decontamination, equipment rinsates and field blanks will not be collected.

Trip Blanks. Trip blanks are used to detect contamination by halogenated VOCs during sample shipping, handling, and analysis. Trip blanks are 40 ml volatile organic compound vials of American Society for Testing and Materials (ASTM) type II water that are filled at the contract laboratory, transported to the site and returned to the lab with the other VOC samples collected. The blanks will be preserved in exactly the same method as the samples (see section IV A). Trip blanks are not to be opened in the field. One trip blank will accompany each cooler containing the VOC samples. The trip blank will only be analyzed for VOCs.

Field Blanks. Field blanks will be collected by the sampling team. Field blanks are collected to evaluate and identify sampling problems or inconsistencies that may be introduced in the field. Field blanks will be collected at the first and last house on each sampling day. Field blanks will be collected using, as a minimum, deionized, distilled water for 218.2 and organic-free water for 524.2. All field blanks will be given a fictitious sample identification number and submitted as blinds to BC Laboratory.

Split Sampling. Pima County Department of Environmental Quality (PDEQ) will complete the analysis and reporting on split samples collected on a minimum of 30% of the total number of VOC samples collected for VOC analysis and a minimum of 30% of the total number of chromium samples collected for Total chromium analysis. The samples will be collected by AL/OEM using the same methods described later in section IV B and C for VOC and IV D and E for chromium samples collected. The samples will be provided to the PDEQ designated representative accompanying the sampling team. PDEQ will provide the means for identifying homes where split samples will be collected, handling, shipping, and analyzing the samples. The laboratory PDEQ selects for the analysis will utilize the same methods (EPA 524.2 for VOCs and EPA 218.2 for total chromium and EPA 7196 for chromium VI) and the same level of quality control including full CLP reporting. On days when split samples are collected, PDEQ will also prepare their own trip blanks to accompany the VOC samples. The split sampling plan will be developed by PDEQ. PDEQ and EPA Region IX will ensure consistency between the two plans. Evaluation of the split sampling results is at the discretion of EPA Region IX.

Field Duplicates. Field duplicate samples will be collected at the first and last house on each sampling day. If the second source to be sampled for VOCs is at an outdoor spigot, that will be the preferred location for a field duplicate. Otherwise, field duplicates will be collected at the tap with all other samples. All field duplicates for total chromium will be collected at the kitchen tap. First, collect VOC samples with duplicates, consecutively, then collect chromium samples with duplicates. Acceptability of duplicate samples analyses will be determined by project goals, as outlined in section III, part B.

#### Field Measurements

Conductivity, pH, and temperature will be measured at every sampling point. These measurements are not compound specific. The instruments will be calibrated and field-checked according to the manufacturers' instructions. This information will be recorded on the calibration

log sheets. Documentation will be discussed later in the plan. The pH meter will be calibrated at the beginning of each days' sampling to a 2-point standard using pH 7.0 and 10.0 calibration standards. In addition, the pH meter will be calibrated at each sample location or whenever the instrument has been turned off. The accuracy of the meter will be +/- 0.5 pH units. The conductivity meter will be calibrated to 1-point in a 0.01M KCl solution. The instructions for the specific pieces of equipment that will be used for the field measurements are in Appendix D.

#### Laboratory Accreditation

BC Laboratories is certified by the state of California, Department of Health Services for EPA Method 524.2. BC is also certified by the State of Arizona, Department of Health Services for EPA 524.2 and 218.2. The American Association for Laboratory Accreditation has also certified BC Labs for all classes of EPA potable water sampling methods. Laboratory certification is an indicator of lab performance, providing the evidence that the lab follows necessary protocols. Appendix A is a summary of BC Lab's accreditation.

#### Detection Limits

BC Lab's practical quantitation limit will be the project goal. The PQLs are listed in Appendix E.

#### Field Quality Control Requirements for VOC Sampling

For VOCs the sampling protocol outlined in "Standard Methods", 17th Edition, section 6010B, Sample Collection and Preservation will be followed. The sampling procedure is provided in Section IV and summarized here. The samples will be collected in 40 ml glass vials with teflon lids. The preservative (HCL) will be added to the bottle prior to sampling to achieve a pH of <2. If the water to be sampled has been chlorinated (as determined by questioning the homeowner and positive responses verified through the use of a standard DPD {N,N-diethyl-p-phenylenediamine} Ferrous Titrimetric Method - SM 4500-CL F.) Three milligrams sodium thiosulfate will be added to the sample container as recommended in SM 6010B for samples collected from chlorinated, potable, water systems to prevent further formation of halogenated hydrocarbons. The sodium thiosulfate will be added by the field sampling crew to the vial prior to sampling only if they determine that the system is chlorinated. Since the samples are collected from private wells, the water will be assumed to be chlorine free unless information is provided to the contrary. The sample will be maintained at 4° C, ± 2° C never frozen. Bubbles are not permitted in the sampling container. The holding time for these samples is 14 days from time of collection.

#### Field Quality Control Requirements for Total Chromium Sampling

The sampling procedures are provided in section IV of this document and summarized here. The sample will be collected in a 1 liter amber bottle or plastic cubetainer. Sufficient nitric acid

will be added to the sampling container prior to sampling to preserve the sample with a pH <2. The holding time for these samples is 6 months from time of collection.

#### Data Validation

Upon receipt of the laboratory analytical results (approximately four to six weeks after the last sample was delivered to the contract lab), US EPA Region IX will conduct a data validation on 100% of the samples following the "National Functional Guidelines for Inorganic and Organic Data Review". Region IX will then provide the final report on the data quality by a date to be determined by US EPA Region IX.

#### Field Documentation

All documentation must be completed in waterproof blue or black ink. Corrections must be marked with a single line, dated and initialed. Hand written documents must be legible. Field documentation for this sampling effort will consist of a logbook, field forms, and sample labels.

#### Logbook

The logbook will be used to chronicle the sampling efforts. It delineates activities occurring on a given sampling day. The sampling team leader must sign and date the logbook at the end of each page. Pages should be numbered and not removed from the document. The logbook should contain specific descriptions of sample locations, purging information, and field measurements. The following items should also be included in the log book:

- (a) Outline of activities for each day, including homes sampled w i t h sample numbers provided.
- (b) Specific comments on problems that occurred during daily activities along with their final resolution.
- (c) Record of phone calls pertaining directly to field sampling activities.
- (d) The airbill numbers of the samples shipped and the chain of custody form details.
- (e) Records of instrument calibration, including the names of personnel performing the calibrations. Also, equipment type and serial number should be recorded.

#### Field Data Forms

A single form will be used for each home sampled. The form will include house address, phone number, sample number, date, time and field screening results. An example of the form that will be used is provided in Figure 1.2.

### Sample Identification

The following information must be recorded on the sample label placed on the sample bag:

- (a) Sample number
- (b) Date/time of sample collection
- (c) Name of sampler
- (d) Sample preservation method
- (e) Type of analysis requested

The sample number will also be written on the sample container label.

### Chain of Custody

Sample possession must be traceable. To track sample possession, an official CC form shall be maintained. The form utilized is provided by the contract lab (Figure 1.3). Custody seals will be used when samples are shipped to the laboratory to ensure that no sample violations occur during transportation. After sample collection, a chain of custody seal will be placed on each sample container as soon as possible after sample collection. Samples will never be left unattended unless they are secured, either in the sampling team's vehicle or lodging facility. Chain of custody will be maintained until the time that the samples are released to the designated air carrier.

## FIELD SAMPLING PROCEDURES

### VOC Sample Preparation

Pre-cleaned 40 ml vials with teflon (TFE) septum and free of volatile organic compounds will be used for the VOC sampling. Prior to collecting the sample(S) for VOC analysis, a single 40 ml sample will be collected from the tap to determine the volume of HCL required to adequately preserve the sample (i.e. to achieve a pH of  $<2$ ). In the initial 40 ml vial, 2 drops of 1:1 HCL will be added prior to sample collection. Once the vial has been filled with the water sample, the pH will be tested. If the pH is not low enough, an additional drop of HCL will be added and the pH tested again. The volume of HCL needed for that test sample (i.e. number of drops) will be used for all subsequent samples collected from that home. The tested vial will be discarded.

### VOC Sample Collection Location

Two samples will be collected from each home. The first sample will be collected from the kitchen cold water tap as a "first draw" sample to provide an estimate of the water quality consumed by the residents. It is assumed that when water is used from the kitchen tap the water is not run for any more time than is needed to reach the appropriate temperature. The second

sample will be collected from one of the following three locations. The locations are ordered by preference:

1. A spigot located between the well head and (assumed) water storage tank.
2. The kitchen sink tap. However, if there is a water treatment system on-line, the second sample must be taken before such a system (i.e. backyard spigot).

The sampling team will make no alterations to the existing piping system at the residence in order to collect the second sample. The second sample is meant to serve as only as an indicator of the aquifer water quality. Prior to collection of the second set of samples, the sampling team will purge the system, measuring the purge flow rate by filling a 1 liter container. It is recognized that the volume of water purged from each second sample location (including heavy water use at the residence prior to sampling) will be a significant determining factor on whether the sample will be representative of aquifer water quality. Purging length will be determined by a marked change in temperature or 10 minutes, whichever comes first. The rationale for using change in temperature as an endpoint is that it is an indicator that water other than that stored in the pipes is being purged. If this is the case at an outdoor spigot, it may be a better indicator of aquifer quality. If indoors, it may draw on the water in the holding tank and present another scenario for possible exposure to contaminants. If the only available sample collection location is the kitchen tap, it will also be used for the second sample, employing a purge as described above.

#### VOC Sample Collection

Samples for VOC analysis will be collected using the following procedures. Any exterior filters, screens, or aeration devices will be removed from the sample location prior to sample collection. No samples will be collected through a rubber hose. The flow will be reduced to approximately 100 ml/min and the sample collected carefully from the flowing stream in such a way as to prevent air bubbles from passing through the sample as the vial is filled. The sample vial should be filled to just overflowing. Leave the water running. Place the sample on a level surface and position the TFE side of the septum seal at the edge of the convex meniscus and gently slide the seal across the vial, against the glass surface. Seal the vial by screwing the plastic cap on tightly. Invert the sample and lightly tap the cap on a solid surface. The absence of entrapped air indicates a successful seal. If bubbles are present, open the vial, and add a few additional drops of water and repeat the sealing procedure. The vial will contain the sample preservative (approximately two drops concentrated HCl), so care must be taken not to let the sample overflow more than is necessary to collect a bubble-free sample. The sample should not be topped off more than twice in attempts to exclude air bubbles as the preservative may be overly diluted. Chill the samples to  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  immediately after collection and hold at that temperature in an atmosphere free of organic solvent vapors until analysis.

### Total Chromium Sample Collection

After the VOC sample is collected from the kitchen tap, the water should be left running, and a one liter acid rinsed plastic or glass container placed under the faucet and a full one liter volume collected. The samples will be preserved by adding nitric acid to the sample bottle. Bubble entrapment is not of concern for the total chromium samples. The bottle will be capped and lightly shaken to mix in the acid. A small quantity of sample will be poured into the bottle cap where the pH will be tested using litmus paper. The pH must be less than or equal to 2. The sample in the cap will be discarded, and the pH of the sample will be adjusted further as necessary. The samples will be chilled to 4 degrees Celsius immediately upon collection and preservation. Only one sample will be collected for total chromium analysis (kitchen tap).

### Laboratory QC Samples

The sampling team will designate for the laboratory which samples to use as a laboratory QC for duplicate and/or matrix spike analyses. The sampling team will identify on the sampling form which samples should be used as laboratory QCs and will target those wells which are known or suspected to be contaminated. The team will provide BC Laboratories a double volume sample for samples designated to be the laboratory QC sample.

### Field Measurement Procedures

The specific methodologies used for the measurement of pH and conductivity of the water samples is provided in Appendix B and C respectively. The information from these measurements plus the water temperature will be recorded on the field data form, Figure 1.2. These results for each residence sample will be provided in addition to the VOC and total chromium results. Field stability parameters will be measured at both sampling locations.

### Sample Packaging

Each sample container will be sealed with custody seal over the cap. A cooler chest will be maintained for the VOC vials and also for the total chromium sample containers. The bottle cap will be labeled with a grease pencil. No marking will be made on the VOC vial to avoid interference with the laboratory's analytical equipment. Each VOC vial or total chromium bottle will be placed in a separate plastic bag, the bag labeled with the sample number and analyte and then placed in the appropriate cooler. The vials will be placed on top of double-bagged ice and covered by additional double-bagged ice. Loose ice will not be poured into the cooler, nor will ice be placed in the chest in its original package. Blue ice may be used in place of double-bagged ice. Suitable packaging material will be placed in the cooler chests to prevent sample bottles from making direct contact with each other or with the coolant packs or ice. The samples collected for VOC analysis will not be permitted to freeze.

### Sample Delivery to Laboratories

The sample team will deliver each days' samples to the designated air carrier for next day delivery to the contract lab. In addition, the team will ensure that the chain of custody form is put in a plastic bag, sealed and taped to the inside top of the cooler lid along with other documentation that needs to be transferred with the samples. Six custody seals will be placed around the cooler lid, signing and dating each seal. The individual who packed the cooler will place a shipping label on the top of the cooler and deliver it to the air freight company at the airport.

## REPORTING SAMPLING AND FIELD ANALYSIS DATA

### Results of Analysis

The results of the water sampling analyses and field measurements will be provided to USEPA Region IX, ATSDR, PDEQ, and the Air Force agencies involved at the TIASS (ASC, AF ANG, AFCEE). The EPA will have the responsibility for providing and/or presenting this information to the individual households. The report will present the method of analysis (including specific analytes), the analytical and field sample results, detection limits, and definitions of the data qualifiers used. The data table in the report will include:

- (1) Address of home sampled
- (2) Date sample collected
- (3) Sample collection location
- (4) Field measurement results
  - (a) Temperature
  - (b) pH
  - (c) Conductivity
- (5) Laboratory analytical results and data qualifiers for all analytes. Samples at the detection limit should be reported with the "U" data qualifier.
  - (a) Halogenated VOC results
  - (b) Total chromium results
  - (c) Duplicate sampling results, if applicable
- (7) Results of most recent, previous, sampling efforts
- (8) Comments particular to the sampling

### Data Validation

The independent data validation will be the responsibility of USEPA Region IX. Data validation will be completed on 100% of the sample results. The results of the data validation will be provided as an attachment to the final report.

### Logs, Field Forms, Chain of Custody Forms

Copies of the chain of custody and field sampling forms for each household will be included as attachments to the report. Copies of logbook pages containing field sampling information will also be included in the final report.

### SAMPLING HEALTH AND SAFETY PLAN

The samples collected under this plan are from private potable water wells. There should not be a concern for contact with the water being sampled. The samples will be collected from the kitchen tap in all cases. Where available, a water sample will be collected between the well head and storage tank. This second sample collection location may present a potential for injury due to electrical/mechanical equipment near the well-head. In addition, there may be other hazards unique to the individual sample location (i.e., sheet metal covering the pump, insects or animals inhabiting the well housing structure). The sample team will be made aware of these potential hazards. The sample team will receive information concerning any particular hazards from the DTD survey team.

Samples will be preserved, as appropriate, with either concentrated hydrochloric or nitric acid. Appropriate personal protective equipment (PPE) (eye protection and latex gloves) should be worn when transferring the acid to individual sampling containers. Extreme care should be taken to prevent the acid from splashing onto the hands or into the eyes during sample collection. Since acid will be present in the sampling containers and to prevent any potential contamination of the sample, latex gloves will be worn during the sampling procedures. Latex gloves should be changed between sampling locations.

Calibration of the pH meter requires the use of standards with a low pH (i.e., pH 4.0). PPE should be used (eye protection and gloves) and care should be exercised when calibrating this piece of equipment to avoid skin and eye contact with the calibration standards.

Material Safety Data Sheets will be maintained for all preservation and calibration chemicals used. The MSDSs will be maintained at the field laboratory facility (tentatively, Davis Monthan AFB, Bioenvironmental Engineering Office). The MSDSs will be available for the sampling team to review prior to the initiation of the sampling effort.

All members of the sampling team will be 40-hour HAZWOPER trained (Hazardous Waste Operators and Emergency Response). In addition, team members will also have received AF Hazard Communication training. Certification of these training efforts is documented on the individual's AF Form 55 (Health and Safety Training Record).

Presented below is a list of key items sampling personnel should be aware of during field activities

1. Sampling will most likely take place during hot weather - dress accordingly to prevent excessive water loss, fatigue, or heat exhaustion.
2. Drink plenty of fluids.
3. Wear shade hat, sunglasses, and sunscreen if sensitive to prolonged sunlight exposures.
4. Always be aware of your limitations and the surroundings of each sample location.

5. Please call 911 for all emergencies. Locations of the nearest hospital will be provided to the sampling team prior to initiating the field work.

AL/OEM will be responsible for ensuring that the sampling team has been provided a health and safety briefing covering potential health and safety concerns unique to the Tucson area and to the sampling locations. This information will be provided in the form of an oral briefing or a written document.

Figure 1  
Area of Concern, TIA Superfund Site

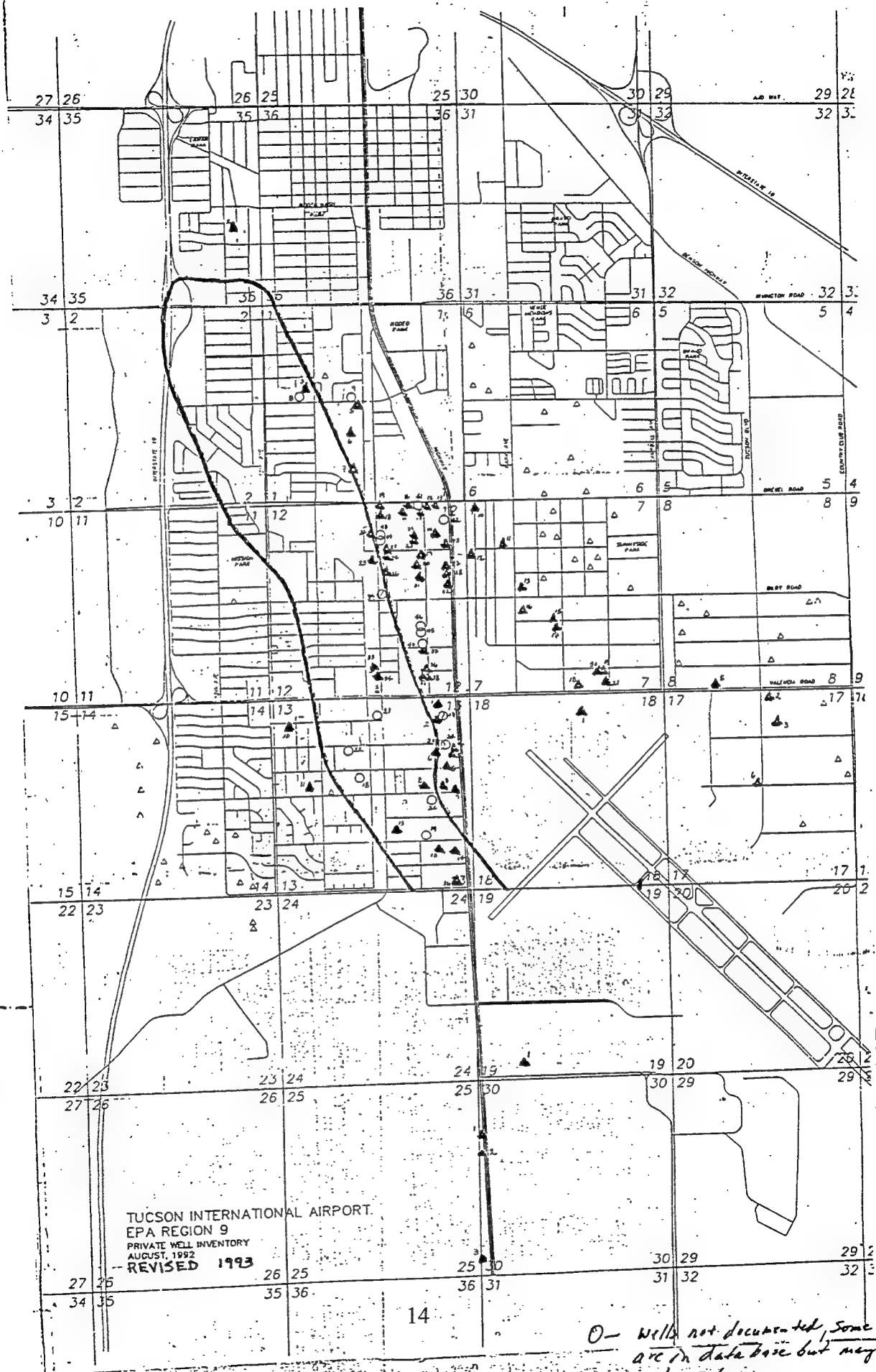


Figure 2  
Field Data Form  
For Use at the TIASS Well Water Sampling Effort

<b>Field Data Form</b> <b>TIA Superfund Site</b> <b>Well Water Sampling Effort</b>
--

Date:	Address:	
Time Start:	Phone:	
Time Stop:	Sampler:	Initials:
Equipment ID:	S/N:	Calibrate:

Field Measurements									
pH/Temp									
Site Measured Values:									
Conductivity									
Site Measured Value:									

Sample #	Method	Detection Limits	Location
GP-94-	524.2	0.5 ppb	
GP-94-	218.2	10 ppb	
GP-94-	524.2	0.5 ppb	
GP-94-			

Figure 3  
Chain of Custody Form  
BC Laboratories, Inc  
For Use at the TIASS Superfund Site Well Water Sampling Effort

# CHAIN OF CUSTODY

4100 Alias Court  
Bakersfield, California 93308

LABORATORIES, INC.



## Report To:

Name:  
Address:  
City:  
State:  
Attn:

Zip:

Phone:

Project:  
Project #:  
Sampler Name:  
Other:

Sample Description

Date & Time Sampled

Matrix (S) Soil (SL) Sludge  
(W) Water (Other)

## Analysis Requested

Samples rec. cold (y/n)  
Custody Seals (y/n)  
Results Needed by:  
Date & Time

Number and  
Container Type

## Comment:

## Billing Info:

Name:  
Address  
City  
Attention:  
Time:  
Miles:  
P.O.#

State

## Sample Disposal

☐ BC Disposal @ 5.00 ea.  
☐ Return to client

Relinquished by: (Signature)  
Received by: (Signature)  
Date: Time:

Relinquished by: (Signature)  
Received by: (Signature)  
Date: Time:

Relinquished by: (Signature)  
Received by: (Signature)  
Date: Time:

Relinquished by: (Signature)  
Received by: (Signature)  
Date: Time:

Relinquished by: (Signature)  
Received by: (Signature)  
Date: Time:

Relinquished by: (Signature)  
Received by: (Signature)  
Date: Time:

# Appendix A

## BC Laboratories Accreditation Summary

**FORWARD**

When Bakersfield Core Laboratory was founded in 1949, "environmental" was just another word in the dictionary. BC Laboratories, Inc., has gradually evolved from an agricultural-petroleum based service into a full service environmental laboratory. Diversified sample matrices ranging from drinking waters to solids and sludges are routinely analyzed for general minerals, metals, and 500-600-8000 series organics. A fully equipped field services department is maintained to complement analytical efforts and extend laboratory capabilities.

BC Laboratories, Inc. has entered the 1990's in a new 18,000 square foot facility specifically designed to assist the quality sector of the analytical process. Departments, 1) wet chemistry, 2) metals, and 3) organics (volatiles, semi-volatiles), are segregated and/or are on separate air systems to reduce inter-laboratory contamination. Refrigerated storage space is in excess of 350 square feet, thus high volumes of samples can be accommodated under preserved conditions. In order to comply with OSHA standards, the laboratory is equipped with a multitude of safety showers, eye wash stations, laboratory hoods (24), fire extinguishers (24), and other protective equipment such as chemical spill kits, fire blankets, and first aid stations. The facility was built from the ground up to be an environmental laboratory.

Within the facility, highly trained personnel work with state-of-the-art instrumentation and an active QA/QC program to provide reliable quality results. Instrumentation includes 5 GC/MS instruments, 15 GC's dedicated to specific analyses, 3 ICAP's, 5 AA's including 2 flameless units, 3 IC's, 6 multi-chemistry auto analyzer systems, 1 ICP/MS, 2 TOX analyzers, a CHONS analyzer and various other equipment necessary to guarantee backup systems to every test run. All employees are on a continuing training program which includes interviewed evaluations done on a semi-annual basis. Constant reinvestment by the ownership has kept BC Laboratories, Inc. current with new instrumentation and advanced analytical technologies.

BC Laboratories, Inc., handles environmental-related type work including: Title 22, SUPERFUND, Groundwater Monitoring, LUFT Manual, Waste Acceptance, and State Drinking Water Standards. Operating under two shifts has allowed for increased capacity and has aided in maintaining acceptable turnaround and holding times. In addition to State of California Accreditation, BC Laboratories, Inc. maintains State of Arizona and New York certifications for out of state work.

Customer service is handled through the Client Services department. Client service representatives are assigned accounts to keep track of work progress, account specific requirements and any difficulties that may occur. These representatives act as customer advocates. Depending on the size of the account, a Project Manager may be assigned to support services.

BC Laboratories Inc., which is A2LA accredited, is striving to become the quality leader of the industry. We supply quality, fully documented products at reasonable cost with extra service. Custom reporting can be done with the aid from a full time LIMS manager, kept on staff to update and maintain a custom Laboratory Information Management System, and modify report requirements or formats. Disk reporting is done routinely. From sampling to final reporting, BC Laboratories will work with you to satisfy your needs.

## DEPARTMENT OF HEALTH SERVICES

151 BERKELEY WAY  
BERKELEY, CA 94704-1011

(510) 540-2800

7/15/93

## Certified Laboratory:

Enclosed is one or more corrected pages of your recently issued Environmental Laboratory Accreditation Program (ELAP) certificate number 1186.

The corrections were made as a result of discovery of typographical errors, or errors of omission. We apologize for any inconvenience these errors may have caused before their discovery and correction.

If you have any further questions, please call us at (510) 540-2800.

Sincerely yours,

William R. Ray  
Water\wastewater Laboratory Consultant  
Environmental Laboratory  
Accreditation Program

enclosures

ENVIRONMENTAL LABORATORY ACCREDITATION/REGISTRATION  
List of Approved Fields of Testing and Analytes

BC Laboratories, Inc.  
4100 Atlas Court  
Bakersfield, CA

TELEPHONE No: (805) 327-4911  
CALIFORNIA COUNTY: Kern

CERTIFICATE NUMBER: 1186  
EXPIRATION DATE: 05-31-94

1 Microbiology of Drinking Water and Wastewater (05-19-90)

1.1	Total Coliforms in Drinking Water by Multiple Tube Fermentation	Y
1.2	Fecal Coliforms/E. Coli in Drinking Water by MTF	Y
1.3	Total Coliforms in Drinking Water by Membrane Filter Technics	N
1.4	Fecal Coliforms/E. Coli in Drinking Water by Membrane Filter Technics	N
1.5	Total Coliforms and E. Coli in Drinking Water by MMO-MUG	Y
1.6	Total Coliforms in Drinking Water by Clark's Presence/Absence	N
1.7	Fecal Coliforms/E. Coli in Drinking Water by Clark's Presence/Absence	N
1.8	Heterotrophic Plate Count	Y
1.9	Total Coliforms in Wastewater by Multiple Tube Fermentation	Y
1.10	Fecal Coliforms in Wastewater by MTF	Y
1.11	Total Coliforms in Wastewater by Membrane Filter Technics	N
1.12	Fecal Coliforms in Wastewater by Membrane Filter Technics	N
1.13	Fecal Streptococci or Enterococci by Multiple Tube Technics	Y
1.14	Fecal Streptococci or Enterococci by Membrane Filter Technics	N

2 Inorganic Chemistry and Physical Properties of Drinking Water excluding Toxic Chemical Elements (05-05-17-90)

2.1	Alkalinity	Y	2.12	Sulfate	Y
2.2	Calcium	Y	2.13	Total Filterable Residue and Conductivity	Y
2.3	Chloride	Y	2.14	Iron (Colorimetric Methods Only)	N
2.4	Corrosivity	Y	2.15	Manganese (Colorimetric Methods Only)	N
2.5	Fluoride	Y	2.16	Phosphate, ortho	Y
2.6	Hardness	Y	2.17	Silica (Colorimetric Methods Only)	N
2.7	Magnesium	Y	2.18	Cyanide	Y
2.8	MBAS	Y			
2.9	Nitrate	Y			
2.10	Nitrite	Y			
2.11	Sodium	Y			

3 Analysis of Toxic Chemical Elements in Drinking Water (05-17-90)

3.1	Arsenic	Y	3.11	Silver	N
3.2	Barium	N	3.12	Zinc	N
3.3	Cadmium	Y	3.13	Aluminum	N
3.4	Chromium, total	N	3.14	Asbestos	N
3.5	Copper	Y	3.15	EPA Method 200.7	Y
3.6	Iron	N	3.16	EPA Method 200.8 (Unregulated Elements and Lead Only)	N
3.7	Lead	Y	3.17	Antimony	Y
3.8	Manganese	N	3.18	Beryllium	N
3.9	Mercury	Y	3.19	Nickel	Y
3.10	Selenium	Y	3.20	Thallium	Y

4 Organic Chemistry of Drinking Water (measurement by GC/MS combination) (05-17-90)

4.1	EPA Method 501.3	N
4.2	EPA Method 524.2	Y
4.3	EPA Method 525	Y
4.4	EPA Method 513	N

5 Organic Chemistry of Drinking Water (excluding measurements by GC/MS combination) (05-17-90)

5.1	EPA Method 501.1	N	5.14	EPA Method 531.1	Y
5.2	EPA Method 501.2	N	5.15	EPA Method 547	Y
5.3	EPA Method 502.1	Y	5.16	EPA Method 548	N
5.4	EPA Method 502.2	Y	5.17	EPA Method 549	N
5.5	EPA Method 503.1	Y	5.18	EPA Method 550	N
5.6	EPA Method 504	Y	5.19	EPA Method 550.1	N
5.7	EPA Method 505	N	5.20	EPA Method 551	N
5.8	EPA Method 506	N	5.21	EPA Method 552	N
5.9	EPA Method 507	Y			
5.10	EPA Method 508	Y			
5.11	EPA Method 508A	N			
5.12	EPA Method 510.1	N			
5.13	EPA Method 515.1	Y			

6 Radiochemistry (-----)

6.1	Gross Alpha and Beta Radiation -----	N
6.2	Total Radium -----	N
6.3	Radium 226 -----	N
6.4	Uranium -----	N
6.5	Radon 222 -----	N
6.6	Radioactive Cesium -----	N
6.7	Iodine 131 -----	N
6.8	Radioactive Strontium -----	N
6.9	Tritium -----	N
6.10	Gamma and Photon Emitters -----	N

6.11	Gross Alpha by Co-precipitation -----	N
6.12	Radium 228 -----	N
6.13	Radioactive Iodine -----	N
6.14	Gross Alpha & Beta in Hazardous Wastes --	N
6.15	Alpha Emitting Radium Isotopes in Haz: Wastes -----	N
6.16	Radium 228 in Hazardous Wastes -----	N

7 Shellfish Sanitation (-----)

7.1	Shellfish meat Microbiology -----	N
7.2	Paralytic Shellfish Poison -----	N
7.3	Domoic Acid -----	N

8 Aquatic Toxicity Bioassays (-----)

8.1	Hazardous Waste Aquatic Toxicity Bioassay (Title 22, CCR, 66261.24(a)(6)) -----	N
8.2	Wastewater Testing According to Kopperdahl (1976) using Freshwater Fish. -----	N
8.3	Wastewater Testing According to EPA/600/4-85/013 using Freshwater and/or Marine Organisms -----	N
8.4	Wastewater Testing by EPA Method 1000.0 -----	N
8.5	Wastewater Testing by EPA Method 1002.0 -----	N
8.6	Wastewater Testing by EPA Method 1003.0 -----	N
8.7	Wastewater Testing by EPA Method 1006 -----	N
8.8	Wastewater Testing by EPA Method 1007 -----	N
8.9	Wastewater Testing by EPA Method 1009 -----	N
8.10	Wastewater Testing According to Anderson, et. al. (1990) using Giant Kelp ( <i>Macrocystis pyrifera</i> ) --	N
8.11	Wastewater Testing According to Anderson, et. al. (1990) using Red Abalone ( <i>Haliotis rufescens</i> ) ---	N
8.12	Wastewater Testing According to Dinnel and Stober (1987) using Purple Sea Urchin ( <i>Strongylocentrotus purpuratus</i> ) -----	N
8.13	Wastewater Testing According to Dinnel and Stober (1987) using Red Sea Urchin ( <i>Strongylocentrotus franciscanus</i> ) -----	N
8.14	Wastewater Testing According to Dinnel and Stober (1987) using Sand Dollar ( <i>Dendraster excentricus</i> ) -----	N
8.15	Wastewater Testing According to procedure E 724-89 (ASTM, 1989) using Pacific Oyster ( <i>Crassostrea gigas</i> ) -----	N
8.16	Wastewater Testing According to procedure E 724-89 (ASTM, 1989) using California Bay Mussel ( <i>Mytilus edulis</i> ) -----	N
8.17	Wastewater Testing According to Standard Methods (APHA, 1989) using an alga ( <i>Skeletonema costatum</i> ) -----	N
8.18	Wastewater Testing According to EPA/600/4-90/027 using Freshwater and/or Marine Organisms -----	N

9 Physical Properties Testing of Hazardous Waste (04-04-88)

9.1	Ignitability by flashpoint determination (Title 22, CCR, 66261.21) -----	Y
9.2	Corrosivity - pH determination (Title 22, CCR, 66261.22) -----	Y
9.3	Corrosivity - Corrosivity towards steel (Title 22, CCR, 66261.22) -----	N
9.4	Reactivity (Title 22, CCR, 66261.23) -----	Y

10 Inorganic Chemistry and Toxic Chemical Elements of Hazardous Waste

10.1	Antimony -----	N
	7040(-----) -----	Y
	7041(04-04-86) -----	Y
10.2	Arsenic -----	N
	7060(05-17-90) -----	Y
	7061(-----) -----	N
10.3	Barium -----	N
	7080(-----) -----	N
	7081(-----) -----	N
10.4	Beryllium -----	N
	7090(-----) -----	Y
	7091(05-17-90) -----	Y
10.5	Cadmium -----	N
	7130(-----) -----	Y
	7131(05-17-90) -----	Y
10.6	Chromium, total -----	N
	7190(-----) -----	Y
	7191(05-17-90) -----	Y
10.7	Cobalt -----	N
	7200(-----) -----	N
	7201(-----) -----	N
10.8	Copper -----	N
	7210(-----) -----	N
	7211(-----) -----	N
10.9	Lead -----	N
	7420(-----) -----	Y
	7421(05-17-90) -----	Y
10.10	Mercury -----	Y
	7470(04-04-86) -----	Y
	7471(04-04-86) -----	Y
10.11	Molybdenum -----	N
	7480(-----) -----	N
	7481(-----) -----	N
10.12	Nickel -----	Y
	7520(04-04-86) -----	Y

10.13	Selenium			10.19	Cyanide		
	7740(04-04-86)	-----	Y		9010(04-04-86)	-----	Y
	7741(04-04-86)	-----	Y	10.20	Fluoride		
10.14	Silver				300.0(-----)	-----	N
	7760(-----)	-----	N		340.1(-----)	-----	N
	7761(04-04-86)	-----	Y		340.2(-----)	-----	Y
10.15	Thallium				340.3(-----)	-----	N
	7840(-----)	-----	N	10.21	Sulfide		
	7841(04-04-86)	-----	Y		9030(04-04-86)	-----	Y
10.16	Vanadium			10.22	Total Organic Lead		
	7910(-----)	-----	N		(03-16-88)	-----	Y
	7911(-----)	-----	N	10.23	EPA Method 6010(03-16-88)	-----	Y
10.17	Zinc			10.24	EPA Method 6020(-----)	-----	N
	7950(-----)	-----	N				
	7951(-----)	-----	N				
10.18	Chromium (VI)						
	7195(-----)	-----	N				
	7196(04-04-86)	-----	Y				
	7197(-----)	-----	N				
	7198(-----)	-----	N				
11	<u>Extraction Tests of Hazardous Waste (04-04-88)</u>						
11.1	California Waste Extraction Test (WET) (Title 22, CCR, 66261.100, Appendix II)	-----	Y				
11.2	Extraction Procedure Toxicity	-----	Y				
11.3	Toxicity Characteristic Leaching Procedure (TCLP) All Classes	-----	Y				
11.4	Toxicity Characteristic Leaching Procedure (TCLP) Inorganics Only	-----	N				
11.5	Toxicity Characteristic Leaching Procedure (TCLP) Extractables Only	-----	N				
11.6	Toxicity Characteristic Leaching Procedure (TCLP) Volatiles Only	-----	N				
12	<u>Organic Chemistry of Hazardous Waste (measurement by GC/MS combination)</u>						
12.1	EPA Method 8240(03-16-88)	-----	Y				
12.2	EPA Method 8250(-----)	-----	N				
12.3	EPA Method 8270(03-16-88)	-----	Y				
12.4	EPA Method 8280(-----)	-----	N				
12.5	EPA Method 8290(-----)	-----	N				
12.6	EPA Method 8260(04-16-93)	-----	Y				
13	<u>Organic Chemistry of Hazardous Waste (excluding measurements by GC/MS combination)</u>						
13.1	EPA Method 8010(04-04-86)	-----	Y	13.13	EPA Method 8310(09-15-93)	-----	Y
13.2	EPA Method 8015(-----)	-----	N	13.14	EPA Method 632 (05-17-90)	-----	Y
13.3	EPA Method 8020(04-04-86)	-----	Y	13.15	Total Petroleum Hydrocarbons		
13.4	EPA Method 8030(-----)	-----	N		(LUFT Manual) (03-16-88)	-----	Y
13.5	EPA Method 8040(-----)	-----	N	13.16	EPA Method 8011(-----)	-----	N
13.6	EPA Method 8060(-----)	-----	N	13.17	EPA Method 8021(04-16-93)	-----	Y
13.7	EPA Method 8080(04-04-86)	-----	Y	13.18	EPA Method 8070(-----)	-----	N
13.8	EPA Method 8090(-----)	-----	N	13.19	EPA Method 8110(-----)	-----	N
13.9	EPA Method 8100(-----)	-----	N	13.20	EPA Method 8141(04-16-93)	-----	Y
13.10	EPA Method 8120(-----)	-----	N	13.21	EPA Method 8330(-----)	-----	N
13.11	EPA Method 8140(05-17-90)	-----	Y				
13.12	EPA Method 8150(03-16-90)	-----	Y				
14	<u>Bulk Asbestos Analysis (-----)</u>						
14.1	1% or Greater Asbestos Concentrations (Title 22, CCR, 66261.24(a)(2)(A)) -----N						
15	<u>Substances Regulated Under the California Safe Drinking Water and Toxic Enforcement Act (Proposition 65) and Not Included in Other listed Groups.</u>						
16	<u>Wastewater Inorganic Chemistry, Nutrients and Demand (-----)</u>						
16.1	Acidity	-----	Y	16.12	Cyanide	-----	Y
16.2	Alkalinity	-----	Y	16.13	Cyanide amenable to Chlorination	-----	Y
16.3	Ammonia	-----	Y	16.14	Fluoride	-----	Y
16.4	Biochemical Oxygen Demand	-----	Y	16.15	Hardness	-----	Y
16.5	Boron	-----	Y	16.16	Kjeldahl Nitrogen	-----	Y
16.6	Bromide	-----	Y	16.17	Magnesium	-----	Y
16.7	Calcium	-----	Y	16.18	Nitrate	-----	Y
16.8	CBOD	-----	Y	16.19	Nitrite	-----	Y
16.9	Chemical Oxygen Demand	-----	Y	16.20	Oil and Grease	-----	Y
16.10	Chloride	-----	Y	16.21	Organic Carbon	-----	Y
16.11	Chlorine Residual, total	-----	Y	16.22	Oxygen, Dissolved	-----	Y

16.23	pH	Y
16.24	Phenols	Y
16.25	Phosphate, ortho-	Y
16.26	Phosphorus, total	Y
16.27	Potassium	Y
16.28	Residue, Total	Y
16.29	Residue, Filterable (TDS)	Y
16.30	Residue, Nonfilterable (TSS)	Y
16.31	Residue, Setttable (SS)	Y
16.32	Residue, Volatile	Y
16.33	Silica	Y
16.34	Sodium	Y
16.35	Specific Conductance	Y
16.36	Sulfate	Y
16.37	Sulfide (includes total & soluble)	Y
16.38	Sulfite	N

16.39	Surfactants (MBAS)	Y
16.40	Tannin and Lignin	N
16.41	Turbidity	Y
16.42	Iron (Colorimetric Only)	N
16.43	Manganese (Colorimetric Only)	N
16.44	Total Recoverable Petroleum Hydrocarbons	Y
16.45	Total Organic Halides	Y

17 Toxic Chemical Elements in Wastewater (05-17-90)

17.1	Aluminum	N
17.2	Antimony	Y
17.3	Arsenic	Y
17.4	Barium	N
17.5	Beryllium	Y
17.6	Cadmium	Y
17.7	Chromium (VI)	Y
17.8	Chromium, total	N
17.9	Cobalt	N
17.10	Copper	Y
17.11	Gold	Y
17.12	Iridium	N
17.13	Iron	N
17.14	Lead	Y
17.15	Manganese	N
17.16	Mercury	Y
17.17	Molybdenum	N

17.18	Nickel	Y
17.19	Osmium	N
17.20	Palladium	N
17.21	Platinum	N
17.22	Rhodium	N
17.23	Ruthenium	N
17.24	Selenium	Y
17.25	Silver	Y
17.26	Strontium	N
17.27	Thallium	Y
17.28	Tin	Y
17.29	Titanium	Y
17.30	Vanadium	Y
17.31	Zinc	N
17.32	EPA Method 200.7	Y
17.33	EPA Method 200.8	N
17.34	DCP	N
17.35	Asbestos	N

18 Organic Chemistry of Wastewater (measurements by GC/MS combination (05-17-90)

18.1	EPA Method 624	Y
18.2	EPA Method 625	Y
18.3	EPA Method 1613	N
18.4	EPA Method 1625	N
18.5	EPA Method 613	N

19 Organic Chemistry of Wastewater (excluding measurements by GC/MS combination) (05-17-90)

19.1	EPA Method 601	Y
19.2	EPA Method 602	Y
19.3	EPA Method 603	N
19.4	EPA Method 604	N
19.5	EPA Method 605	N
19.6	EPA Method 606	N
19.7	EPA Method 607	N
19.8	EPA Method 608	Y
19.9	EPA Method 609	N
19.10	EPA Method 610	N
19.11	EPA Method 611	N
19.12	EPA Method 632	Y
19.13	EPA Method 619	N

20 Inorganic Chemistry and Toxic Chemical Elements of Pesticide Residues in Food (-----)

20.1	Processed Foods by One of the Following Methods	
	Atomic Absorption Spectrophotometry	N
	Inductively Coupled Plasma Atomic Emission Spectrophotometry	N
	Inductively Coupled Plasma/Mass Spectrometry	N
	Colorimetry	N
20.2	Raw Commodities by One of the Following Methods	
	Atomic Absorption Spectrophotometry	N
	Inductively Coupled Plasma Atomic Emission Spectrophotometry	N
	Inductively Coupled Plasma/Mass Spectrometry	N
	Colorimetric	N
20.3	Dairy Products by One of the Following Methods	
	Atomic Absorption Spectrophotometry	N
	Inductively Coupled Plasma Atomic Emission Spectrophotometry	N
	Inductively Coupled Plasma/Mass Spectrometry	N
	Colorimetry	N

20.4	Feed Products by One of the Following Methods	
	Atomic Absorption Spectrophotometry .....	N
	Inductively Coupled Plasma Atomic Emission Spectrophotometry .....	N
	Inductively Coupled Plasma/Mass Spectrometry .....	N
	Colorimetry .....	N
21	<u>Organic Chemistry of Pesticide Residues in Food (measurements by GC/MS) (-----)</u>	
21.1	Gas Chromatographic/Mass Spectrometric Methods in Processed Foods .....	N
21.2	Gas Chromatographic/Mass Spectrometric Methods in Raw Commodities .....	N
21.3	Gas Chromatographic/Mass Spectrometric Methods in Dairy Products .....	N
21.4	Gas Chromatographic/Mass Spectrometric Methods in Feed Products .....	N
22	<u>Organic Chemistry of Pesticide Residues in Food (Excluding Measurement by GC/MS Combination)</u> (-----)	
22.1	Halogenated Compounds in Processed Foods by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.2	Organophosphorous Compounds in Processed Foods by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.3	Carbamates in Processed Foods by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.4	Halogenated Compounds in Raw Commodities by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.5	Organophosphorous Compounds in Raw Commodities by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.6	Carbamates in Raw Commodities by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.7	Halogenated Compounds in Dairy Products by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.8	Organophosphorous Compounds in Dairy Products by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.9	Carbamates in Dairy Products by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.10	Halogenated Compounds in Feed Products by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.11	Organophosphorous Compounds in Feed Products by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N
22.12	Carbamates in Feed Products by One of the Following Methods	
	Gas Chromatography .....	N
	High Pressure Liquid Chromatography .....	N
	Liquid Chromatography/Mass Spectrometry .....	N

DIVISION OF STATE LABORATORY SERVICES  
OFFICE OF LABORATORY LICENSURE AND CERTIFICATION

ling Number: AZ0345

Date: April 26, 1993

Facility: B.C. LABORATORIES, INC.

Director: DANIEL K. SCHULTZ

Address: 4100 ATLAS Ct.  
City: BAKERSFIELD  
Zip Code: 93308

County: KERN  
Telephone: (805) 327-4911

State: CA  
Ext:

\*\*\* LIST OF LICENSED PARAMETERS AND APPROVED METHODS BY PROGRAM \*\*\*

BILLING NUMBER	PROGRAM	PARAMETER	APPROVED METHOD
AZ0345		ATOMIC ABSORPTION SPECTROPHOTOMETER	AA SPEC. #3
		ATOMIC ABSORPTION SPECTROPHOTOMETER	AA SPEC. #4
		ATOMIC ABSORPTION SPECTROPHOTOMETER	AA SPECTROPH.
		ATOMIC ABSORPTION SPECTROPHOTOMETER	AA SPEC. #2
		GAS CHROMATOGRAPH	GAS CHRO. #2
		GAS CHROMATOGRAPH	GAS CHRO. #4
		GAS CHROMATOGRAPH	GAS CHROMATOGR.
		GAS CHROMATOGRAPH	GAS CHRO. #3
		GAS CHROMATOGRAPH/MASS SPECTROMETER	GC/MS #4
		GAS CHROMATOGRAPH/MASS SPECTROMETER	GC/MS #2
		GAS CHROMATOGRAPH/MASS SPECTROMETER	GC/MS
		GAS CHROMATOGRAPH/MASS SPECTROMETER	GC/MS #3
		HIGH PERFORMANCE LIQUID CHROMATOGRAPH	HPLC #2
		HIGH PERFORMANCE LIQUID CHROMATOGRAPH	HPLC
		INDUCTIVELY COUPLED PLASMA SPECTROMETER	ICP #2
		INDUCTIVELY COUPLED PLASMA SPECTROMETER	ICP SPECTROPH.
AZ0345	HW	ALUMINUM	EPA 6010
		ANTIMONY	EPA 6010
		ANTIMONY	EPA 7041
		AROMATIC VOLATILES	EPA 8020
		ARSENIC	EPA 7061
		ARSENIC	EPA 7060
		BARIUM	EPA 6010
		BERYLLIUM	EPA 7091
		BERYLLIUM	EPA 6010
		CADMIUM	EPA 6010
		CADMIUM	EPA 7131
		CALCIUM	EPA 6010

PROGRAMS:

R=AIR, HW=HAZARDOUS WASTE

W=SAFE DRINKING WATER, WW= WASTEWATER

BILLING NUMBER	PROGRAM	PARAMETER	APPROVED METHOD
AZ0345	HW	CALCIUM	EPA 7140
		CHLORIDE	EPA 9251
		CHLORINATED HERBICIDES	EPA 8150
		CHROMIUM TOTAL	EPA 6010
		CHROMIUM TOTAL	EPA 7191
		CHROMIUM (VI)	EPA 7196
		COBALT	EPA 6010
		COPPER	EPA 6010
		CORROSIVITY pH	EPA 9040
		CORROSIVITY TOWARDS STEEL	EPA 1110
		CYANIDE TOTAL AND AMENABLE	EPA 9012
		FUEL CLASS HYDROCARBONS	BLS-191
		HALOGENATED VOLATILES	EPA 8010
		IGNITABILITY (FLASH POINT)	EPA 1010
		IRON	EPA 6010
		LEAD	EPA 7421
		LEAD	EPA 6010
		MAGNESIUM	EPA 7450
		MAGNESIUM	EPA 6010
		MERCURY	EPA 7470
		MERCURY	EPA 7471
		MOLYBDENUM	EPA 6010
		NICKEL	EPA 6010
		NITRATE	EPA 9200
		NONHALOGENATED VOLATILES	EPA 8015
		ORGANOCHLORINE PESTICIDES	EPA 8080
		ORGANOPHOSPHORUS PESTICIDES	EPA 8140
		pH SOIL	EPA 9045
		POLYNUCLEAR AROMATIC HYDROCARBONS	EPA 8310
		POTASSIUM	EPA 6010
		POTASSIUM	EPA 7610
		REACTIVITY	REACTIVITY
		SELENIUM	EPA 7740
		SELENIUM	EPA 7741
		SELENIUM	EPA 6010
		SEMIVOLATILE COMPOUNDS	EPA 8270
		SILVER	EPA 6010
		SODIUM	EPA 7770
		SPECIFIC CONDUCTANCE	EPA 9050
		STRONTIUM	EPA 6010
		SULFATE	EPA 9036
		SULFIDE	EPA 9030
		THALLIUM	EPA 7841
		THALLIUM	EPA 6010
		TOTAL ORGANIC CARBON	EPA 9060
		TOTAL ORGANIC HALIDES	EPA 9020
		TOXICITY CHARACTERISTIC LEACHING PROCEDURE	EPA 1311
		VANADIUM	EPA 6010

PROGRAMS:

AR=AIR, HW=HAZARDOUS WASTE

DW=SAFE DRINKING WATER, WW= WASTEWATER

BILLING NUMBER	PROGRAM	PARAMETER	APPROVED METHOD
AZ0345	HW	VANADIUM	EPA 7911
		VOLATILE COMPOUNDS	EPA 8240
		VOLATILE COMPOUNDS	EPA 8260
		ZINC	EPA 6010
AZ0345	SDW	ACID AND BASE/NEUTRAL COMPOUNDS	EPA 525
		ALKALINITY	EPA 310.1
		AROMATIC VOLATILES	EPA 502.2
		ARSENIC	EPA 206.2
		BARIUM	EPA 200.7
		CADMIUM	EPA 213.2
		CADMIUM	EPA 200.7
		CALCIUM	EPA 215.1
		CALCIUM	EPA 215.2
		CARBAMATES	EPA 531.1
		CHLORIDE	EPA 300.0
		CHLORIDE	EPA 325.2
		CHLORINATED PESTICIDES	EPA 508
		CHLOROPHENOXY HERBICIDES	EPA 515.1
		CHROMIUM TOTAL	EPA 200.7
		CHROMIUM TOTAL	EPA 218.2
		COLOR	EPA 110.3
		COPPER	EPA 220.2
		COPPER	EPA 200.7
		CORROSIVITY	SM 2330B
		EDB AND DBCP	EPA 504
		FLUORIDE	EPA 340.2
		HALOGENATED VOLATILES	EPA 502.2
		HARDNESS	SUM EPA 215&242
		HYDROGEN ION (pH)	EPA 150.1
		IRON	EPA 200.7
		LEAD	SM 3113B
		LEAD	EPA 200.7
		MAGNESIUM	EPA 242.1
		MANGANESE	EPA 200.7
		MANGANESE	SM 3113B
		MERCURY	EPA 245.1
		MERCURY	EPA 245.2
		NITRATE	SM 4500-NO3 F
		NITRATE	EPA 353.2
		NITRATE	EPA 300.0
		NITROGEN AND PHOSPHORUS PESTICIDES	EPA 507
		POLYCHLORINATED BIPHENYLS	EPA 508
		RESIDUE FILTERABLE (TDS)	EPA 160.1
		SELENIUM	EPA 270.2

PROGRAMS:

IR=AIR, HW=HAZARDOUS WASTE

SD=SAFE DRINKING WATER, WW= WASTEWATER

BILLING NUMBER	PROGRAM	PARAMETER	APPROVED METHOD
A20345	SDW	SELENIUM	SM 3114C
		SILVER	EPA 200.7
		SILVER	EPA 272.2
		SODIUM	EPA 273.1
		STRONTIUM	EPA 200.7
		SULFATE	EPA 375.2
		SULFATE	EPA 300.0
		SURFACTANT	SM 5540C
		SURFACTANT	EPA 425.1
		TURBIDITY	EPA 180.1
		VOLATILE ORGANICS	EPA 524.2
		ZINC	EPA 200.7
A20345	WW	ACID AND BASE/NEUTRAL COMPOUNDS	EPA 625
		ACIDITY	EPA 305.1
		ALKALINITY	EPA 310.1
		ALUMINUM	EPA 200.7
		AMMONIA	EPA 350.1
		AMMONIA	SM 4500-NH3 B
		ANTIMONY	EPA 200.7
		AROMATIC VOLATILES	EPA 602
		ARSENIC	EPA 206.2
		ARSENIC	SM 3114B
		BARIUM	EPA 200.7
		BERYLLIUM	EPA 200.7
		BORON	EPA 200.7
		BROMIDE	EPA 300.0
		CADMIUM	SM 3120B
		CADMIUM	EPA 213.2
		CADMIUM	EPA 200.7
		CALCIUM	EPA 215.1
		CALCIUM	SM 3120B
		CARBAMATES	EPA 632
		CHLORIDE	EPA 300.0
		CHROMIUM TOTAL	EPA 200.7
		CHROMIUM TOTAL	EPA 218.2
		CHROMIUM (VI)	SM 3500-CR D
		COBALT	EPA 200.7
		COLOR	EPA 110.2
		COPPER	EPA 220.2
		COPPER	EPA 200.7
		CYANIDE	EPA 335.3
		CYANIDE	EPA 335.2
		FLUORIDE	EPA 340.2
		GOLD	EPA 231.2

PROGRAMS:

IR=AIR, HW=HAZARDOUS WASTE

DW=SAFE DRINKING WATER, WW= WASTEWATER

BILLING NUMBER	PROGRAM	PARAMETER	APPROVED METHOD
AZ0345	WW	HALOGENATED VOLATILES	EPA 601
		HARDNESS	EPA 215.1&242.1
		HARDNESS	EPA 130.2
		HARDNESS	EPA 200.7, CA&MG
		HYDROGEN ION (pH)	EPA 150.1
		IRON	EPA 200.7
		KJELDAHL NITROGEN	EPA 351.2
		LEAD	EPA 239.2
		LEAD	EPA 200.7
		MAGNESIUM	EPA 242.1
		MANGANESE	EPA 200.7
		MERCURY	EPA 245.1
		MOLYBDENUM	EPA 200.7
		NICKEL	EPA 249.2
		NICKEL	EPA 200.7
		NITRATE	EPA 353.2
		NITRATE	EPA 300.0
		NITRITE	EPA 354.1
		NITRITE	SM 4500-NO2 B
		NITRITE	EPA 353.2
		OIL AND GREASE	EPA 413.1
		ORGANOCHLORINE PESTICIDES	EPA 608
		PHENOLS	EPA 420.2
		PHOSPHATE ORTHO	EPA 365.1
		PHOSPHORUS TOTAL	EPA 365.1
		POLYCHLORINATED BIPHENYLS	EPA 608
		POLYNUCLEAR AROMATICS	EPA 610
		POTASSIUM	EPA 258.1
		RESIDUE FILTERABLE (TDS)	EPA 160.1
		RESIDUE NONFILTERABLE (TSS)	EPA 160.2
		RESIDUE SETTLEABLE	EPA 160.5
		RESIDUE TOTAL	EPA 160.3
		RESIDUE VOLATILE	SM 160.4
		SELENIUM	EPA 270.2
		SELENIUM	SM 3113B
		SILICA	EPA 200.7
		SILVER	EPA 200.7
		SILVER	EPA 272.2
		SODIUM	EPA 273.1
		SPECIFIC CONDUCTANCE	EPA 120.1
		STRONTIUM	EPA 200.7
		SULFATE	EPA 300.0
		SULFATE	SM 4500-SO4 B
		SULFATE	EPA 375.2
		SULFIDE	EPA 376.2
		SULFIDE	EPA 376.1
		SURFACTANTS (MBAS)	EPA 425.1
		THALLIUM	EPA 200.7

PROGRAMS:

IR=AIR, HW=HAZARDOUS WASTE

OH=SAFE DRINKING WATER, WW= WASTEWATER

BILLING NUMBER	PROGRAM	PARAMETER	APPROVED METHOD
AZ0345	WW	THALLIUM	SM 3113B
		THALLIUM	EPA 279.2
		TIN	EPA 282.2
		TOTAL ORGANIC CARBON	SM 5310
		TOTAL ORGANIC CARBON	EPA 415.2
		TURBIDITY	EPA 180.1
		VANADIUM	EPA 200.7
		VOLATILE ORGANICS	EPA 524.1
		ZINC	EPA 200.7

PROGRAMS:

AIR=AIR, HW=HAZARDOUS WASTE

DW=SAFE DRINKING WATER, WW= WASTEWATER



THE AMERICAN  
ASSOCIATION FOR  
LABORATORY  
ACCREDITATION

## ACCREDITED LABORATORY

A2LA has accredited

**B C LABORATORIES, INC.,** BAKERSFIELD, CA

for technical competence in the field of

ENVIRONMENTAL TESTING

The accreditation covers the specific tests and types of tests listed on the agreed scope of accreditation. This laboratory meets the requirements of ISO/IEC guide 25-1990 "General Requirements for the Competence of Calibration and Testing Laboratories" (equivalent to relevant requirements of the ISO 9000 series of standards) and any additional program requirements in the identified field of testing

Presented this 27TH day of JULY 1992



*John W. Locke*

President  
For the Accreditation Council

Certificate Number 0292-01

Valid to JUNE 30, 1994



# American Association For Laboratory Accreditation

## SCOPE OF ACCREDITATION

B C LABORATORIES, INC.  
4100 Atlas Court  
Bakersfield, CA 93308  
Anthony Bonanno Phone: 805 327 4911

## ENVIRONMENTAL

Valid To: June 30, 1994

Certificate Number: 0292-01

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

### Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Methylene Blue Active Substances, Microbiology, Misc.- Electronic Probes (pH, F<sup>-</sup>, O<sub>2</sub>), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide, Turbidity

Potable Water: metals, nutrients, demands, classical (wet) chemistry, microbiology, purgeable organics, extractable organics, pesticides-herbicides-PCB's

Nonpotable Water: metals, nutrients, demands, classical (wet) chemistry, microbiology, purgeable organics, extractable organics, pesticides-herbicides-PCB's

Solid/Hazardous Waste: metals, nutrients, demands, classical (wet) chemistry, purgeable organics, extractable organics, pesticides-herbicides-PCB's and hazardous waste characteristics (ignitability, conductivity, corrosivity, reactivity, Paint Filter Liquids Test, EP toxicity, and TCLP)

Further details about the laboratory's accreditation, including its capability in terms of specific analytes, are available upon request.



# American Association For Laboratory Accreditation

## SUPPLEMENT TO SCOPE OF ACCREDITATION

B C LABORATORIES  
4100 Atlas Court  
Bakersfield, CA 93308  
Anthony Bonanno Phone: 805 327 4911

### ENVIRONMENTAL

Valid as of: July 27, 1992  
Valid until: June 30, 1994

Certificate Number: 0292-01

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform recognized EPA methods for the following determinations:

#### Potable Water

Metals: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Au, Fe, Pb, Mg, Mn, Hg, Mo, Ni, K, Se, Si, Ag, Na, Tl, Sn, Ti, V, Zn

Nutrients: Ammonia (as N), Kjeldahl nitrogen, Nitrate-nitrite (as N), Nitrite (as N), Orthophosphate (as P), Total phosphorus

Demands: COD, TOC

Classical Chemistry: Acidity, Alkalinity, Bromide, Chloride, Color, Cyanide, Cyanide amenable to chlorination, Fluoride, Hardness, pH, MBAS, Oil and grease, Dissolved oxygen, Phenols, Total residue, Filterable residue, Nonfilterable residue, Settleable residue, Volatile residue, Specific conductance, Sulfate, Sulfide, Surfactants, Turbidity, Corrosivity-calc.carb. stability

Microbiology: Fecal coliform, Total coliform

Purgeable Organics: Benzene, Bis(2-chloroethoxy)methane, Bis(2-chloroisopropyl)ether, Bromobenzene, Bromodichloromethane, Bromoform, Bromomethane, n-Butylbenzene, sec-Butylbenzene, tert-Butylbenzene, Carbon tetrachloride, Chlorobenzene, Chloroethane, 2-Chloroethylvinyl ether, Chloroform, Chloromethane, Chlorotoluene, Dibromochloromethane, 1,2-Dibromo-3-chloropropane (DBCP), Dibromomethane, 1,2-Dibromoethane (EDB), 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, Dichlorodifluoromethane, 1,1-Dichloroethane, 1,2-Dichloroethane, 1,1-Dichloroethene, cis-1,2-Dichloroethene, trans-1,2-Dichloroethene, 1,2-Dichloropropane, 1,3-Dichloropropane, 2,2-Dichloropropane, 1,1-Dichloropropene, cis-1,3-Dichloropropene, trans-1,3-Dichloropropene, Ethylbenzene, Hexachlorobutadiene, Isopropylbenzene, 1,4-Isopropyltoluene, Idomethane, Methylene Chloride, Naphthalene, n-propylbenzene, Styrene,

1,1,1,2-Tetrachloroethane, 1,1,2,2-Tetrachloroethane, Tetrachloroethene, Toluene, 1,1,1-Trichloroethane, 1,1,2-Trichloroethane, Trichloroethene, Trichlorofluoromethane, Trichloropropane, 1,2,3-Trichloropropane, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Trihalomethanes, Vinyl chloride, Xylene total, 1,2-Xylene, 1,3-Xylene, 1,4-Xylene

Extractable Organics: Acenaphthene, Acenaphthylene, Acetophenone, Aniline, Anthracene, Benzidine, Benzoic acid, Benzo(a)anthracene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(ghi)perylene, Benzo(a)pyrene, Benzyl alcohol, Bis(2-chloroethoxy)methane, Bis(2-chloroethyl)ether, Bis(2-chloroisopropyl)ether, Bis(2-ethylhexyl)phthalate, 4-Bromophenylphenyl ether, Butyl benzyl phthalate, 2-sec-Butyl-4,6-dinitrophenol (DNBP), 4-Chloro-3-methylphenol, 1-Chloronaphthalene, 2-Chloronaphthalene, 2-Chlorophenol, 4-Chlorophenylphenyl ether, Chrysene, Cresols (methyl phenols), 2-Cyclohexyl-4,6-dinitrophenol, Dibenzo(a,h)anthracene, Dibenzofuran, 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, 3,3'-Dichlorobenzidine, 2,4-Dichlorophenol, 2,6-Dichlorophenol, Diethylphthalate, 2,4-Dimethylphenol, Dimethylphthalate, Di-n-butylphthalate, Di-n-octylphthalate, Dinitrobenzene, 2,4-Dinitrophenol, 2,4-Dinitrotoluene, 2,6-Dinitrotoluene, Diphenylamine, 1,2-Diphenylhydrazine, Fluoranthene, Fluorene, Hexachlorobenzene, Hexachlorobutadiene, Hexachlorocyclopentadiene, Hexachloroethane, Indeno(1,2,3-cd)pyrene, Isophorone, 2-Methyl-4,6-Dinitrophenol, 2-Methylnaphthalene, 2-Methylphenol, 4-Methylphenol, Naphthalene, 2-Naphthylamine, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, Nitrobenzene, 2-Nitrophenol, 4-Nitrophenol, N-Nitrosodimethylamine, N-Nitrosodi-n-propylamine, N-Nitrosodiphenylamine, Pentachlorophenol, Phenanthrene, Phenol, Pronamide, Pyrene, 1,2,4-Trichlorobenzene, Trichlorophenols, 2,4,5-Trichlorophenol, 2,4,6-Trichlorophenol

Pesticides-herbicides-PCBs: Aldrin, Atrazine, alpha-BHC, beta-BHC, delta-BHC, gamma-BHC (Lindane), Chlordane (technical), 2,4-D, Dalapon, 2,4-DB, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Diazinon, Dicamba, Dieldrin, Dinoseb, Diuron, Endosulfan I (alpha), Endosulfan II (beta), Endosulfan sulfate, Endrin, Endrin aldehyde, Heptachlor, Heptachlor epoxide, Kepone, Methiocarb, Methoxychlor, PCB-1016 (arochlor), PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1254, PCB-1260, Simazine, 2,4,5-T, 2,4,5-TP (Silvex), Toxaphene

#### Nonpotable Water

Metals: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Mo, Ni, K, Se, Si, Ag, Na, Tl, Sn, Ti, V, Zn

Nutrients: Ammonia (as N), Kjeldahl nitrogen, Nitrate-nitrite (as N), Nitrite (as N), Orthophosphate (as P), Total phosphorus

Demands: BOD, Carbonaceous BOD

Classical Chemistry: Chloride, Cl (residual), Fluoride, Hardness, pH, Fe, Mg, Mn, MBAS, Oil and Grease, Sulfide, Sulfite, Surfactants, Temperature, Turbidity, Zn, Corrosivity-calc.carb. stability

Microbiology: Fecal coliform, Total coliform, Fecal streptococci

Purgeable Organics: Acetone, Acetonitrile, Acrolein, Acrylamide, Acrylonitrile, Benzene, Bis(2-chloroethoxy)methane, Bis(2-chloroisopropyl)ether, Bromobenzene, Bromodichloromethane, Bromoform, Bromomethane, 2-Butanone, n-Butylbenzene, sec-Butylbenzene, tert-Butylbenzene, Carbon disulfide, Carbon tetrachloride, Chlorobenzene, Chloroethane, 2-Chloroethylvinyl ether, Chloroform, Chloromethane, Chlorotoluene, Dibromochloromethane, 1,2-Dibromo-3-chloropropane (DBCP), Dibromomethane, 1,2-Dibromoethane (EDB), 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, Dichlorodifluoromethane, 1,1-Dichloroethane, 1,2-Dichloroethane, 1,1-Dichloroethene, cis-1,2-Dichloroethene, trans-1,2-Dichloroethene, 1,2-Dichloropropane, 1,3-Dichloropropane, 2,2-Dichloropropane, 1,1-Dichloropropene, cis-1,3-Dichloropropene, trans-1,3-Dichloropropene, Diethyl ether, Ethanol, Ethylbenzene, Ethyl methacrylate, 2-Hexanone, Hexachlorobutadiene, Isopropylbenzene, 1,4-Isopropyltoluene, Idomethane, Methylene Chloride, Methyl ethyl ketone, Methyl isobutyl ketone, 4-Methyl-2-pentanone, Naphthalene, n-propylbenzene, Styrene, 1,1,1,2-Tetrachloroethane, 1,1,2,2-Tetrachloroethane, Tetrachloroethene, Toluene, 1,1,1-Trichloroethane, 1,1,2-Trichloroethane, Trichloroethene, Trichlorofluoromethane, Trichloropropane, 1,2,3-Trichloropropane, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Vinyl acetate, Vinyl chloride, Xylene total, 1,2-Xylene, 1,3-Xylene, 1,4-Xylene

Extractable Organics: Acenaphthene, Acenaphthylene, Acetophenone, Aniline, Anthracene, Benzo(a)anthracene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(ghi)perylene, Benzo(a)pyrene, Benzyl alcohol, Bis(2-chloroethoxy)methane, Bis(2-chloroethyl)ether, Bis(2-chloroisopropyl)ether, Bis(2-ethylhexyl)phthalate, 4-Bromophenylphenyl ether, Butyl benzyl phthalate, 2-sec-Butyl-4,6-dinitrophenol (DNBP), 4-Chloro-3-methylphenol, 1-Chloronaphthalene, 2-Chloronaphthalene, 2-Chlorophenol, 4-Chlorophenylphenyl ether, Chrysene, Cresols (methyl phenols), 2-Cyclohexyl-4,6-dinitrophenol, Dibenzo(a,h)anthracene, Dibenzofuran, 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, 3,3'-Dichlorobenzidine, 2,4-Dichlorophenol, 2,6-Dichlorophenol, Diethylphthalate, 2,4-Dimethylphenol, Dimethylphthalate, Di-n-butylphthalate, Di-n-octylphthalate, Dinitrobenzene, 2,4-Dinitrophenol, 2,4-Dinitrotoluene, 2,6-Dinitrotoluene, Diphenylamine, 2,6-Diphenylhydrazine, 1,2-Diphenylhydrazine, Fluoranthene, Fluorene, Hexachlorobenzene, Hexachlorobutadiene, Hexachlorocyclopentadiene, Hexachloroethane, Indeno(1,2,3-cd)pyrene, Isophorone, 2-Methyl-4,6-Dinitrophenol, 2-Methylnaphthalene, 2-Methylphenol, 4-Methylphenol, Naphthalene, 2-Naphthylamine, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, Nitrobenzene, 2-Nitrophenol, 4-Nitrophenol, N-Nitrosodimethylamine, N-Nitrosodi-n-propylamine, N-Nitrosodiphenylamine, Pentachlorophenol, Phenanthrene, Phenol, Pyrene, 1,2,4-Trichlorobenzene, Trichlorophenols, 2,4,5-Trichlorophenol, 2,4,6-Trichlorophenol

Pesticides-herbicides-PCBs: Aldrin, Atrazine, Azinphos methyl, alpha-BHC, beta-BHC, delta-BHC, gamma-BHC (Lindane), Bolstar, Chlordane (technical), Chloropropham, Chlorpyrifos, Coumaphos, 2,4-D, Dalapon, 2,4-DB, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Demeton-O, Demeton-S, Diazinon, Dicamba, Dichlorvos, Dichloroprop, Dicofol, Dieldrin, Dinoseb, Disulfoton, Diuron, Endosulfan I (alpha), Endosulfan II (beta), Endosulfan sulfate, Endrin, Endrin aldehyde,

Ethion, Ethoprop, Fensulfothion, Fenthion, Fenuron, Fenuron-TCA, Heptachlor, Heptachlor epoxide, Kepone, Linuron, Malathion, Merphos, Methoxychlor, Mevinphos, Mexacarbate, Monuron, Monuron TCA, Neburon, Parathion ethyl, Parathion methyl, PCB-1016 (arochlor), PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1254, PCB-1260, Phorate, Promethryn, Propham, Propoxur, Secbumeton, Siduron, Simazine, Stiropfos (Tetrachlorvinphos), Swep, 2,4,5-T, Tokuthion (Prothiofos), 2,4,5-TP (Silvex), Toxaphene, Trichloronate

Solid Waste/Hazardous Waste

Metals: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Mo, Ni, K, Se, Si, Ag, Na, Tl, Sn, Ti, V, Zn

Nutrients: Ammonia (as N), Nitrate-nitrite (as N), Total phosphorus

Demands: TOX

Classical Chemistry: Alkalinity, Chloride, Cyanide, Cyanide amenable to chlorination, Fluoride, Oil and grease, Phenols, Sulfide stability

Purgeable Organics: Acetone, Acetonitrile, Acrolein, Acrylamide, Acrylonitrile, Benzene, Bis(2-chloroethoxy)methane, Bis(2-chloroisopropyl)ether, Bromobenzene, Bromodichloromethane, Bromoform, Bromomethane, 2-Butanone, n-Butylbenzene, sec-Butylbenzene, tert-Butylbenzene, Carbon disulfide, Carbon tetrachloride, Chlorobenzene, Chloroethane, 2-Chloroethylvinyl ether, Chloroform, Chloromethane, Chlorotoluene, Dibromochloromethane, 1,2-Dibromo-3-chloropropane (DBCP), Dibromomethane, 1,2-Dibromoethane (EDB), 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, Dichlorodifluoromethane, 1,1-Dichloroethane, 1,2-Dichloroethane, 1,1-Dichloroethene, cis-1,2-Dichloroethene, trans-1,2-Dichloroethene, 1,2-Dichloropropane, 1,3-Dichloropropane, 2,2-Dichloropropane, 1,1-Dichloropropene, cis-1,3-Dichloropropene, trans-1,3-Dichloropropene, Diethyl ether, Ethanol, Ethylbenzene, Ethyl methacrylate, 2-Hexanone, Hexachlorobutadiene, Isopropylbenzene, 1,4-Isopropyltoluene, Idomethane, Methylene Chloride, Methyl ethyl ketone, Methyl isobutyl ketone, 4-Methyl-2-pentanone, Naphthalene, n-propylbenzene, Styrene, 1,1,1,2-Tetrachloroethane, 1,1,2,2-Tetrachloroethane, Tetrachloroethene, Toluene, 1,1,1-Trichloroethane, 1,1,2-Trichloroethane, Trichloroethene, Trichlorofluoromethane, Trichloropropane, 1,2,3-Trichloropropane, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Vinyl acetate, Vinyl chloride, Xylene total, 1,2-Xylene, 1,3-Xylene, 1,4-Xylene

Extractable Organics: Acenaphthene, Acenaphthylene, Acetophenone, Aniline, Anthracene, Benzidine, Benzoic acid, Benzo(a)anthracene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(ghi)perylene, Benzo(a)pyrene, Benzyl alcohol, Bis(2-chloroethoxy)methane, Bis(2-chloroethyl)ether, Bis(2-chloroisopropyl)ether, Bis(2-ethylhexyl)phthalate, 4-Bromophenylphenyl ether, Butyl benzyl phthalate, 2-sec-Butyl-4,6-dinitrophenol (DNBP), 4-Chloro-3-methylphenol, 1-Chloronaphthalene, 2-Chloronaphthalene, 2-Chlorophenol, 4-Chlorophenylphenyl ether, Chrysene, Cresols (methyl phenols), 2-Cyclohexyl-4,6-dinitrophenol, Dibenzo(a,h)anthracene, Dibenzofuran, 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene,

3,3'-Dichlorobenzidine, 2,4-Dichlorophenol, 2,6-Dichlorophenol, Diethylphthalate, 2,4-Dimethylphenol, Dimethylphthalate, Di-n-butylphthalate, Di-n-octylphthalate, Dinitrobenzene, 2,4-Dinitrophenol, 2,4-Dinitrotoluene, 2,6-Dinitrotoluene, Diphenylamine, 1,2-Diphenylhydrazine, Fluoranthene, Fluorene, Hexachlorobenzene, Hexachlorobutadiene, Hexachlorocyclopentadiene, Hexachloroethane, Indeno(1,2,3-cd)pyrene, Isophorone, 2-Methyl-4,6-Dinitrophenol, 2-Methylnaphthalene, 2-Methylphenol, 4-Methylphenol, Naphthalene, 2-Naphthylamine, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, Nitrobenzene, 2-Nitrophenol, 4-Nitrophenol, N-Nitrosodimethylamine, N-Nitrosodi-n-propylamine, N-Nitrosodiphenylamine, Pentachlorophenol, Phenanthrene, Phenol, Pyrene, Tetrachlorobenzenes, 1,2,4,5-Tetrachlorobenzene, 2,3,4,5-Tetrachlorophenol, 2,4,6-Tribromophenol, 1,2,4-Trichlorobenzene, Trichlorophenols, 2,4,5-Trichlorophenol, 2,4,6-Trichlorophenol

Pesticides-herbicides-PCBs: Aldrin, Atrazine, Azinphos methyl, alpha-BHC, beta-BHC, delta-BHC, gamma-BHC (Lindane), Bolstar, Chlordane (technical), Chloroprotham, Chlorpyrifos, Coumaphos, 2,4-D, Dalapon, 2,4-DB, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Demeton-O, Demeton-S, Diazinon, Dicamba, Dichlorvos, Dichloroprop, Dicofol, Dieldrin, Dinoseb, Disulfoton, Diuron, Endosulfan I (alpha), Endosulfan II (beta), Endosulfan sulfate, Endrin, Endrin aldehyde, Ethion, Ethoprop, Fensulfothion, Fenthion, Fenuron, Fenuron-TCA, Heptachlor, Heptachlor epoxide, Kepone, Linuron, Malathion, Merphos, Methoxychlor, Mevinphos, Mexacarbate, Monuron, Monuron TCA, Neburon, Parathion ethyl, Parathion methyl, PCB-1016 (arochlor), PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1254, PCB-1260, Phorate, Promethryn, Protham, Propoxur, Sebumeton, Siduron, Simazine, Stirophos (Tetrachlorvinphos), Swep, 2,4,5-T, Tokuthion (Prothiofos), 2,4,5-TP (Silvex), Toxaphene, Trichloronate

Hazardous Waste Characteristics: Conductivity, Corrosivity, EP Toxicity, Ignitability, Paint Filter Liquids Test, Liquid Release Test, Reactivity, TCLP

Appendix B.  
Field Measurement Instructions  
Temperature and pH

# Measurement of Temperature and pH of Water Samples

## 1. Definitions:

- a. pH: Hydrogen ion ( $H^+$ ) concentration of a solution; the greater the hydrogen ion concentration, the more acidic the solution.
- b. Buffer Solutions: Solutions of essentially stable pH. These solutions contain both a weak acid and its conjugate weak base; the pH changes only slightly on addition of acid or base.
- c. Flow-through cell: A closed container made of inert material with connections and outlets for hoses and measuring probes.

## 2. Procedure:

- a. Check the pH meter before taking it to the field. Visually inspect the instrument, examining all wires and probes for damage. Check the glass bulb at the end of the pH electrode to ensure that it is not broken.
- b. Prepare the electrode for use. Fill the electrode with the correct filling solution according to the manufacturer's operating instructions. Close or cover the filling hole to keep the solution from spilling. If using a liquid junction electrode, ensure that the level of filling solution is always above the reference junction. The filling hole should always be open when the electrode is in use. To use the electrode, remove it from the storage solution and rinse it well with deionized water, being careful to avoid rubbing the electrode or disrupting/scratching the delicate glass bulb on the bottom of the electrode. Connect the pH electrode to the meter.
- c. Calibrate the meter. Use the appropriate calibration buffers to calibrate the meter (i.e., 4.00 and 7.00 if the sample is anticipated to have a pH less than 7.0 or if the sample is greater than 7.0 use the buffer solutions with pH of 7.00 and 10.00). Refer to the manufacturer's operating instructions for the exact calibration procedures. Rinse the electrode with deionized water. Recalibrate the instrument each time it is turned on or at each sampling location. Record the buffers used and the date and time of calibration in the sample logbook.
- d. Measure the pH and temperature of the water sample. Thoroughly rinse a beaker with sample water and place the pH and temperature probes into the beaker. Pour in a fresh sample and allow the readings to stabilize. Read and record the temperature and pH of the sample. In addition, record the time of sampling in the logbook and on the field data form. Rinse the electrodes thoroughly with deionized water and when done sampling, store them according to manufacturer's recommendation.

## Appendix C

### Field Measurement Instructions

### Conductivity

## Measurement of Conductivity of Water Samples

### 1. Definitions:

a. Electrical conductivity. The property of a substance that describes its ability to transfer electricity; the reverse of resistivity.

### 2. Procedure:

a. Inspect the conductivity meter before taking it into the field. Since several different instruments are available for measuring conductivity, each with different features, it is important to read the user's manual to become acquainted with the instrument. Examine all wires, cables, and probes for damage and replace if necessary.

b. Check the instrument against known conductivity standards, preferably employing two standards that bracket the expected conductivity of the sample. Rinse the electrical conductivity probe with deionized water. Measure and record the temperature and conductivity of the standards. Correct the readings to 25 degrees C, if necessary, with the following equation:

$$\text{Conductivity at 25 Degrees C} = \frac{\text{Conductivity at T} \times \text{Cell Constant}}{1 + 0.0191 (T - 25 \text{ deg})}$$

If the instrument has an automatic temperature compensating (ATC) mode, the above calculation is not necessary. When calibrating the instrument, the conductivity standards should fall within  $\pm 10\%$  of the given values. If not, the instrument user's manual should be consulted. After calibrating, rinse the probe with deionized water.

c. Measure the conductivity of the sample. Thoroughly rinse a beaker with sample water. Pour in the fresh sample and immerse the probes in the sample and allow the readings to stabilize. Record the time, electrical conductivity and temperature on the field data form and in the sample log book. Rinse the probes with deionized water and store when completed.

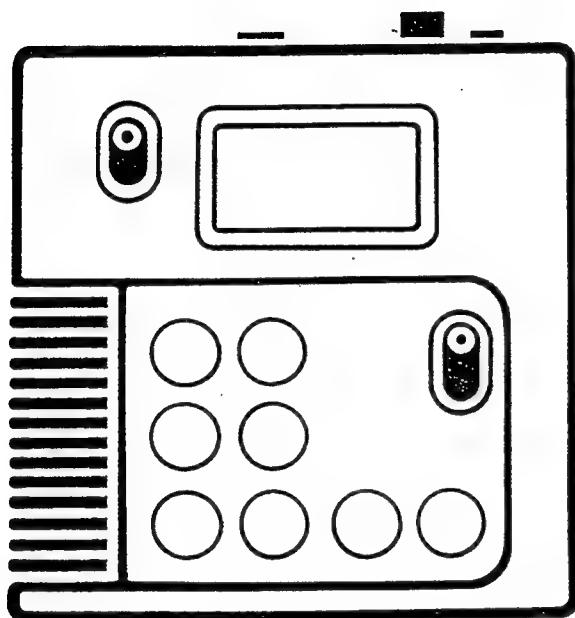
## Appendix D

# Field Sampling Instruments Instructions

# ORION

Orion Research Incorporated  
Laboratory Products Group

## SA 250 pH METER INSTRUCTION MANUAL



## Meter Check Out Procedure

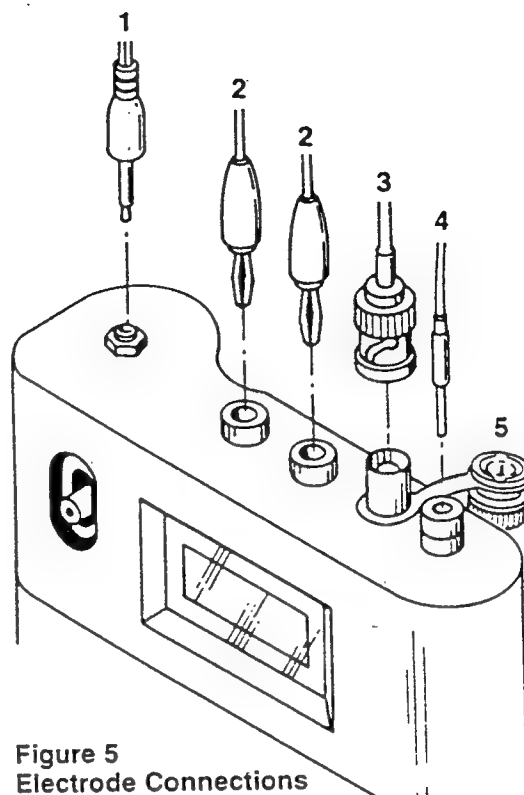
1. Slide power switch to ON position. Attach BNC Shorting Plug (Orion Cat. No. 090045) to BNC connector on top of meter. Refer to **Figure 5**.
2. If using optional AC line converter, connect it to meter and appropriate power source. Proceed to step 4.
3. If **LO BAT** indicator on LCD remains on, the battery must be replaced.
4. Slide mode switch to mV. Display should read  $0 \pm 0.3$ .
5. Slide mode switch to temp. Display should read **25.0**. If **25.0** is not displayed, scroll, using  $\wedge$ ,  $\vee$ , and X10 keys, until **25.0** is displayed and press enter.
6. Slide mode switch to pH .01. Press iso. Display should read the letters **ISO** then a value of **7.00**. If **7.00** is not displayed, scroll until **7.00** is displayed and press enter.
7. Press slope. Display should read the letters **SLP** then a value of **100.0**. If **100.0** is not displayed, scroll until **100.0** is displayed and press enter.
8. Press sample. Observe the letters **pH** then a steady reading of  $7.00 \pm 0.02$  should be obtained. If not, press cal and scroll until **7.00** is displayed and press enter. Press sample and observe a reading of **7.00**.
9. Remove the shorting plug. After a successful completion of steps 1-8 the meter is ready to use with an electrode.

## Electrode Connections

Refer to **Figure 5**.

1. Attach electrodes with BNC connectors to sensor input by sliding connector onto input, pushing down and turning clockwise to lock into position. Connect reference electrodes with pin tip connectors by pushing connector straight into reference input.

**NOTE:** If using a combination electrode with a BNC connector, the reference pin-tip jack is not used (4 in Figure 5).



**Figure 5**  
**Electrode Connections**

### Legend

- 1 AC line converter to line converter jack
- 2 ATC plugs to ATC jacks
- 3 BNC connector to sensor input (shown with shorting plug disconnected)
- 4 Reference pin-tip plug to reference input
- 5 BNC shorting plug (Orion Cat. No. 090045)

## pH Measurements

See Figure 6.

A calibration with one or two buffers should be performed before pH is measured. It is recommended that a calibration with two buffers be performed at the beginning of each day to determine the correct slope of the electrode. This serves the dual purpose of determining if the electrode is working properly and storing the slope value in the meter's memory. Perform a one buffer calibration every two hours to compensate for electrode drift.

Check the stored value for **ISO** before calibration. Unless the isopotential point of the electrode is known verify that the display reads **7.00**. If not, scroll until **7.00** is displayed and press enter. See **Isopotential Point**.

There are two ways of calibrating the SA 250 Meter, autocalibration or manual calibration.

**NOTE: It is recommended to select either autocalibration or manual calibration and not use a combination of the two methods. Following is a description and instructions for each method.**

### Autocalibration

Autocalibration is a feature of the SA 250 Meter that automatically recognizes the 7.00, 4.01 and 10.01 buffers with a range of  $\pm 0.5$  pH units. The user waits until the pH display is stable and presses enter. The SA 250 Meter automatically calibrates to the correct buffer value using temperature compensation. Do not scroll when using autocalibration.

While calibrating, the SA 250 Meter compares actual values to theoretical values to determine if the buffer is within range. Buffers greater than  $\pm 0.5$  pH units from the correct value will trigger an operator assistance code.

It is recommended that an ATC probe be used for autocalibration. If an ATC probe is not used, all samples and buffers should be at the same temperature or use manual temperature compensation. See **Temperature Mode**.

### Autocalibration With Two Buffers

1. Connect electrode(s) to meter. Slide the mode switch to either pH .1 or pH .01. Choose either 4.01 and 7.00, or 7.00 and 10.01 buffers, whichever will bracket your expected sample range.
2. Place electrode(s) into either 4.01, 7.00 or 10.01 buffer.
3. Press cal. The display will alternate between .1 and the pH value of the buffer, indicating this is the first buffer and a value has not been entered. Wait for a stable pH display and press enter. The correct display will freeze for 3 seconds then advance to .2, indicating the meter is ready for the second buffer.

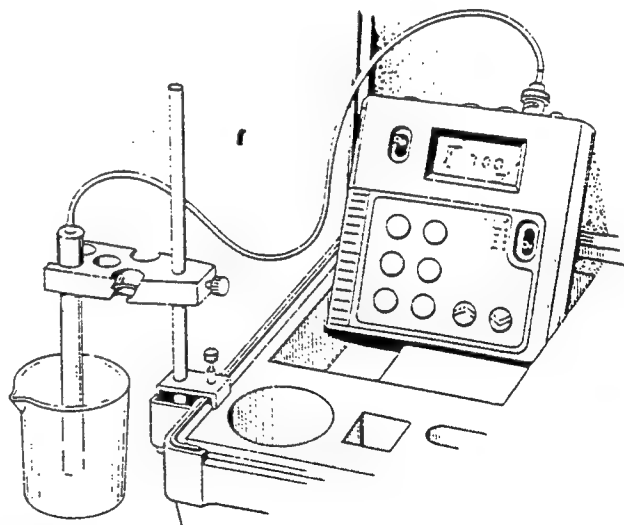
4. Rinse electrode(s) and place into a second buffer. Wait for a stable pH display and press enter. After the second buffer value has been entered the letters **PH** will be displayed. The meter is now calibrated and automatically advances to sample mode.
5. Rinse electrode(s), place into sample. Record pH directly from the meter's display.

### Autocalibration With One Buffer

1. Check slope term by pressing slope. If necessary, scroll and enter the correct value. If slope value is unknown, either enter 100.0 or perform a two buffer calibration. A single buffer calibration does not change the slope term.
2. Connect electrode(s) to meter. Slide mode switch to either pH .1 or pH .01.
3. Place electrodes into either 4.01, 7.00 or 10.01 buffer.
4. Press cal. The display will alternate between .1 and the pH value of the buffer, indicating this is the first buffer and a value has not been entered.
5. Wait for a stable pH reading and press enter.

After enter is pressed the correct display will freeze for 3 seconds then advance to .2, indicating the meter is ready for the second buffer. By pressing sample the letters **PH** will be displayed, indicating the meter has advanced into the sample mode.

6. Rinse electrode(s) and place into sample. Read the pH directly from the display.



**Figure 6**  
**Optional Way to Set Up SA 250 Meter**  
**for Sample Measurements**

## Isopotential Point

The isopotential point is the pH at which the potential (mV) of the electrode will not vary with temperature.

For the majority of pH electrodes the isopotential point is pH 7.00. There are some exceptions where the operating range used for a particular electrode is primarily at one end of the pH scale.

If your pH electrode has an isopotential point other than 7.00, the correct value may be entered as follows:

1. Slide mode switch to pH .1 or pH .01.
2. Press iso.
3. Scroll, using  $\wedge$ ,  $\vee$ , or X10 keys, until correct value is displayed.
4. Press enter.

A two buffer calibration should be performed after an isopotential point value has been changed. It is good practice to verify the isopotential point whenever the meter has been turned on.

## Temperature Mode

Sliding the mode switch to temp will display the temperature in °C. When the temperature is outside of the operating range - 5.0 to 105.0°C, an operator assistance code will be displayed, **E-1** for below - 5°C, or **E 1** for above 105°C.

During a calibration or sample measurement, the mode switch can be changed to temp. When an ATC probe is connected the temperature can be monitored and automatic temperature compensation will take place.

To use manual temperature compensation:

1. Using a thermometer accurate to  $\pm 1^\circ\text{C}$  determine the temperature of the solutions to be measured.
2. Slide mode switch to temp.
3. Scroll, using  $\wedge$ ,  $\vee$  or X10 keys, until the correct temperature value is displayed.
4. Press enter.
5. Return mode switch to either pH .1 or pH .01.

When an ATC probe is not connected, the last entered value of temperature is displayed. If a temperature value has not been entered since the removal of an ATC probe, a default value of 25°C is displayed.

## Potentiometric Measurements

Potentiometric titrations are performed in mV mode using either pH, ion-selective or redox electrodes. Detailed instructions for any ORION Electrode are given in the electrode instruction manual. Titration instructions are included in ORION Redox Electrode (Model 96-78 or 97-78) Instruction Manual, or in standard analytical texts. Electrodes that have a U.S. Standard Connector need a U.S. Standard to BNC Adaptor which are available from Orion (Cat. No. 090033).

## Dissolved Oxygen Measurements

Dissolved oxygen measurements are displayed in ppm  $\text{O}_2$  when ORION Model 97-08 Dissolved Oxygen Electrode is used with ORION SA 250 Meter. Follow these instructions for calibrating the electrode.

1. Connect the Model 970899 to meter and leave electrode mode switch "off".
2. Unplug and do not use an ATC probe.
3. Set the mode switch of the SA 250 Meter to temp and scroll in 25.0°C, press enter.
4. Set the mode switch to pH .1 or pH .01.
5. Press the slope key. Scroll until the value **100.0** appears and press enter.
6. Press the iso key and verify that it is **7.00**. If not, scroll in the value **7.00** and press enter.
7. Press the cal key. Scroll in the value **7.00** and press enter.
8. Press sample.
9. Turn the mode switch on the electrode to BT CK. Good battery operation is indicated by a reading of **13.00** or greater on the meter.
10. Turn the mode switch on the electrode to ZERO. Use the zero calibration control on the electrode to set the meter to read **0.00**.
11. Insert the reservoir (funnel) into a BOD sample bottle containing enough water to just cover the bottom. Insert the electrode, making sure that the electrode tip is *not* immersed in the water and does not have water droplets clinging to the outside of the membrane. Let stand approximately 30 minutes to ensure water saturation of air in BOD bottle. This bottle should be used for electrode storage between measurements.
12. Turn the electrode mode switch to the AIR position. If measurements are being made at sea level, use the AIR calibration control on the electrode to set the pH meter reading to the prevailing barometric pressure in mm Hg (divided by 100). If the barometric pressure is unknown, if the elevation is above sea level or if the sample has a salinity greater than 2 parts per thousand, consult **Table 1** found in the Model 97-08 Instruction Manual to obtain the correct AIR setting.
13. Turn electrode mode switch to  $\text{H}_2\text{O}$  for sample analysis.

Appendix E  
BC Laboratories Standard Operating Procedures  
and Quality Goals

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR VOLATILE ORGANICS  
STANDARD OPERATING PROCEDURES  
EPA METHODS 524.2/624/8240

## 1.0 SCOPE AND APPLICATION

1.1 These methods are used to determine volatile organic compounds in a variety of matrices. Method 624 is used for low level waters. Method 524.2 is used for drinking waters. Method 8240 is used for waste waters and solid waste matrices.

1.2 These methods can be used to quantify most volatile organic compounds that have boiling points below 200 C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in these methods, however for more soluble compounds, the quantitation limits are considerably higher due to poor purging efficiency.

1.3 The practical quantitation limits (PQL's) for these methods vary from matrix to matrix, but are initially 5.0 ug/kg for soils and solid wastes (method 8240) and 0.5 ug/l for waters (methods 524.2 and 624). PQL's will be proportionately higher for samples requiring dilution or reduced sample size in order to avoid saturation of the detector.

1.4 These methods are based upon a purge-and-trap, gas-chromatographic/mass spectrometric (GC/MS) procedure.

## 2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection (in limited applications). The components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information. The chromatographic conditions, as well as typical mass spectrometer operating parameters will be given later.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is extracted with pesticide grade methanol or polyethylene glycol to dissolve the organic constituents. A portion of the solution is then diluted in water and analyzed by the normal water method.

2.3 The purge-and-trap process: Helium is bubbled through the solution at room temperature, and the volatile components are quantitatively transferred from the aqueous phase to the vapor phase. The vapor is passed through a sorbent column where the organic compounds are trapped. After purging is completed, the sorbent trap is heated and backflushed with helium to desorb the components onto the gas chromatographic column. The gas

chromatographic column is heated to elute the compounds, which are detected with the mass spectrometer.

### 3.0 INTERFERENCES

3.1 Interferences purged or extracted from the samples will vary considerably from source to source, depending upon the particular sample or extract being analyzed. The analytical system, however, is checked to ensure freedom from interferences, under the analysis conditions, by the analysis of a method blank.

3.2 Samples can be contaminated by diffusion of volatile organics (especially methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A field blank is prepared from reagent water free of contaminants and carried through the sampling and handling to serve as a check on such contamination.

3.3 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially. Therefore whenever highly concentrated samples are analyzed a system blank immediately follows in order to ensure no cross contamination occurs. The purge-and-trap system may require several such blanks in order to clean the system of such contamination. The section of the laboratory in which volatile analyses takes place is isolated from extractibles in order to minimize solvent contamination of samples by organic vapor from extractions.

### 4.0 APPARATUS AND MATERIALS

4.1 Microsyringes: 10-ul, 25ul, 100ul, 250ul, 500ul, 1000ul are readily available and should only be used for VOA std. prep and dilution.

4.2 Balance: An analytical balance accurate to .10gm for sample weighings is used.

4.3 VOA vials: Used as sampling containers and dilution vials are used.

4.4 Volumetric flasks, Spatulas, Disposable pipets and bulbs are readily available for sample preparation.

4.5 Purge-and-trap: A Tekmar LSC-2 coupled with an ALS are used.

4.5.1 The purging chamber is a 25ml sparging vessel. The sparging needle must be no more than 5mm from the bottom of the sparging vessel. The Helium sparging gas must pass through

the water or solid waste as finely divided bubbles having a diameter of no more than 3mm at their origin.

4.5.2 The trap utilized is a Tekmar trap 25cm in length and 1/8in. in diameter. Starting with the inlet the trap is packed with 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. Prior to daily use the trap is conditioned for approximately 20 minutes with Helium gas backflushing.

#### 4.6 Gas chromatograph/mass spectrometer system:

4.6.1 Gas chromatograph: The gas chromatograph utilized is a Hewlett Packard model 5890 series II.

4.6.2 Column: The column used is a Restek 105M x .53mm RTx 502.2 megabore column.

4.6.2 Mass spectrometer: The mass spectrometer utilized is a Hewlett Packard model 5970B.

4.6.3 Data System: The data system utilized is a Hewlett Packard 1000 RTE-A series with version F software. The GC/MS and data system are capable of meeting all EPA criteria for GC/MS volatile analysis work.

#### 5.0 REAGENTS

5.1 Stock solutions: All stock solutions are purchased as 200ppm in methanol certified standard mixes from various vendors.

5.2 Surrogate standards: The surrogate standards used are 1,2-dichloroethane (d4), Toluene (d8), and Bromofluorobenzene, and are purchased as neat standards and diluted to 50ppm in pesticide grade methanol by the analyst.

5.3 Internal standards: The internal standards used are Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene (d5), and are purchased as neat standards and diluted to 50ppm in pesticide grade methanol by the analyst.

5.4 4-Bromofluorobenzene: A standard solution is prepared from the neat standard in methanol at a concentration of 25ppm for mass calibration checking purposes.

5.5 Calibration standards: The calibration standards are prepared as follows. A 100ml volumetric flask is filled with reagent grade water, proven to be free of any volatile contaminants, and cooled to approximately 20 C. The stock standard solutions are then introduced to the flask in 20ul aliquots each. Making the initial calibration standard at a

concentration of 40ppb in water, and containing all constituents to be analyzed. The initial calibration standard is discarded after use and prepared daily.

5.6 Matrix spiking standard: The matrix spike standard is prepared from neat standards to contain: 1,1-dichloroethene, t-1,2-dichloroethene, 1,2-dichloroethane, carbon tetrachloride, 1,2-dichloropropane, 1,1,2-trichloroethane, tetrachloroethene, chlorobenzene, ethylbenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene in methanol at a concentration of 50ppm.

5.7 Great care must be taken to maintain the integrity of all standard solutions. Therefore all standards are kept in the GC/MS freezer below -10 C and are removed only for daily and initial calibration standard preparation, for a minimum of time, and replaced. An eye must be kept on the response factors of the gaseous compounds. These compounds will be the first to degrade and must be replaced more often than the other standards, typically 3-4 weeks in time.

5.8 Reagent grade water: Defined as water that is free from interferences at the method detection limit for all compounds of interest. The reagent grade water is tested every morning as a method blank. If any interfering compounds are found, the cause of the contamination must be found and corrected before analysis continues.

#### 6.1 Initial calibration for purge-and-trap analysis

##### 6.1.1 GC/MS operating conditions

Electron energy:	70 volts
Mass range:	45-260 amu.
A/D samples:	8
Initial column temperature:	35 C
Initial column hold time:	4 min.
column temp. prog.:	5 C/min.
Final column temperature:	190 C
Final column hold time:	4 min.
Injector temperature:	200 C
Interface temperature:	220 C
Source temperature:	200 C
Carrier gas:	UHP Helium

6.1.2 First daily the GC/MS system must meet the ion-abundance criteria for a 50ng injection of BFB. Analysis cannot begin without meeting these criteria. (tunvoa)

##### 6.1.3 Prepare a 25ml method blank by using a 25ml gastight

syringe and introducing 5ul of internal/surrogate standard mix directly into the syringe. Load the blank into the appropriate sparging container and analyze. Any interferences must be corrected and another reagent water method blank must be analyzed.

6.1.4 The initial calibration standard is prepared by filling a 100ml. volumetric flask with reagent water and cooling to approximately 20 C. 20ul of each of the stock calibration solutions is then added to make a 40ppb initial standard in water. The initial calibration procedure constitutes the making of 5 calibration runs of varying concentration and defining the working range of the instrument. These calibration runs are created by performing the analysis on a 2ml, 5ml, 10ml, 15ml, 20ml aliquot of the initial calibration standard diluted to 25ml final volume in the 25ml gastight syringe. The aliquot sizes relate to sample concentrations of 3.2ug/l, 8ug/l, 16ug/l, 24ug/l, and 32ug/l of each constituent. The internal/surrogate standard solution is added to every blank, spike, standard, and sample to be analyzed by this method by introducing 5ul of the standard into the gastight syringe just before introduction into the sparging chamber.

6.1.5 The average response factor (RF) is calculated by the data system for each compound. The system performance check compounds or (SPCC's) are checked for a minimum average response factor. The minimum average (RF) is .300 and .250 for bromoform. These compounds are checked in order to verify system cleanliness and performance. Active sites in transfer lines or injector port will cause compound instability and degradation yielding low average (RF's).

6.1.6 Using the RF's from the initial calibration, the %RSD is calculated for calibration check compounds CCC's. The %RSD for each CCC MUST be below 30%. This criteria must be met before the initial calibration is to be used.

### 6.3 Daily GC/MS calibration

6.3.1 Prior to the analysis of any sample 50ng of BFB must be injected and the mass spectra must meet the EPA requirements for ion abundances.

6.3.2 The initial calibration curve for each compound of interest must be checked and verified. This is accomplished by analyzing a calibration standard that is at the midrange concentration of the working range (16ug/l). The SPCC and CCC criteria set forth previously must be met.

6.3.3 System performance check compounds: All SPCC's must meet the criteria set forth previously. Some possible problems are standard mixture degradation, injector port liner contamination,

contamination at the front end of the analytical column, and active sites in the column or the chromatographic system.

6.3.4 Calibration check compounds: After the system performance check is met, CCC's are used to check the validity of the initial calibration. The %difference in the response factors using the average RF from the initial curve and the RF from the continuing calibration run. If any percent difference is greater than 20% this is considered a warning limit. If the percent difference for all CCC's is less than 25% the initial calibration is assumed to be valid. If this criteria is not met corrective action must be taken to correct any problems and a new 5-point calibration MUST be generated.

6.3.5 The internal standard responses and retention times in the continuing calibration standard must be checked immediately after data acquisition. If the retention time of any internal standard changes by more than 30 sec. from the last continuing calibration check the chromatographic system must be checked for problems and corrections must be made as required. If the total area for the quantitation ion of any internal standard varies by more than  $\pm 50\%$  from the last daily calibration standard run, the mass spectrometer must be inspected for problems and corrections must be made as appropriate.

#### 6.4 GC/MS analysis

##### 6.4.1 Water analysis (624/524.2/8240):

6.4.1.1 Screening of the sample by visible inspection is necessary in order to provide guidance on whether a sample will need to be diluted before initial analysis. Use good judgement, if a sample appears to be highly contaminated it is better to start with a high dilution and bring the dilution down than to overload the purge-and-trap device and necessitate the running of reagent water blanks to clean the system.

6.4.1.2 All samples must be allowed to warm to room temperature before analysis

6.4.1.3 Use all methods necessary to run the mass spectrometer in the appropriate conditions.

6.4.1.4 BFB tuning ion abundance criteria must be met each day.

6.4.1.5 The Helium purge flow must be maintained at or just below 40ml/min.

6.4.1.6 Analysis of a sample involves opening a 40ml VOA vial and removing the plunger from the syringe and pouring an appropriate amount of the sample into the syringe. The volume in the syringe is adjusted by inverting the syringe and forcing out

any air bubbles and adjusting the plunger to the required volume. If the sample requires additional analysis it is preferred that the second VOA vial be opened as the first's integrity was degraded upon opening.

6.4.1.7 The following procedure is appropriate for the dilution of purgeable samples. All steps must be performed without any delays until the sample is in a gas tight syringe.

6.4.1.7.1 Dilutions may be made in volumetric flasks. Select the appropriate size to make the correct dilution.

6.4.1.7.2 Fill the volumetric with approximately enough water to allow enough room for the amount of sample needed to be added.

6.4.1.7.3 Inject the proper aliquot of sample from a syringe prepared as above. Aliquots of less than 250ul are not recommended. Dilute the sample to the mark with reagent water. Cap the flask, invert, and shake three times. Repeat the above procedure for further dilutions.

6.4.1.7.4 Fill a 25ml syringe with the diluted sample as described above.

6.4.1.8 Add 5ul of internal/surrogate standard to the 25ml syringe with a 25ul gastight syringe.

6.4.1.9 Attach the 25ml syringe to the appropriate sparging chamber and load the sample.

6.4.1.10 Close the sparging chamber valve, set up the data system to make an acquisition, and purge the sample for 11 min.. When the sample is finished purging the LSC-2 will proceed to the desorb ready mode and heat the trap to 175 C. Once this temperature is reached the 6-port valve will switch and backflush the trap at 180 C onto the GC column, and start the GC/MS run. Upon completion of the desorb step the LSC-2 will bake for 32 min. while backflushing the trap with more helium.

6.4.1.11 If upon initial analysis of a sample or a dilution of a sample a concentration of analyte greater than the initial calibration range is found, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated or very high ions from a compound, this analysis must be followed by a reagent water blank. If the blank analysis is not free from contamination, the system must be decontaminated. (ie Running of more blanks or system bakeout)

6.4.1.12 Matrix spike analysis is carried out by selecting a sample which seems to be free from contaminants, and adding 5ul of spiking standard to the sample and analysis as a sample.

## 6.4.2 Sediment/soil and waste samples

6.4.2.1 For an assumed clean sample weigh out 5 gr. of sample directly into a sparging chamber and attach to the appropriate sparging position.

6.4.2.2 Remove the plunger from a 25ml gastight syringe and fill with reagent grade water. Adjust the volume in the syringe to 25mls. and add 5ul of internal/surrogate standard to the syringe. Attach the syringe to the appropriate sparging chamber and introduce the water into the sparging chamber.

6.4.2.3 Analyze the sample according to the methods described above.

6.4.2.4 If any analytes concentration is above the initial calibration range this sample must be diluted.

6.4.2.4.1 A soil/solid waste sample must be diluted in either methanol or polyethylene glycol. The limiting factor to determine the diluent is the purged volume. A maximum of 50ul of methanol can be purged and a maximum of 1.0ml of polyethylene glycol can be purged. Also, Oil samples are best diluted in polyethylene glycol.

6.4.2.4.2 A certain amount of diluent is pipetted into a VOA vial (ie. 5-10mls.) then into this vial is weighed an appropriate amount of sample in order to achieve the dilution desired.

6.4.2.4.3 A desired amount of the diluent is then added to a 25ml syringe, internal/surrogate standard is added and introduced to the proper sparging chamber and analyzed as previously outlined.

## 6.5 Data interpretation

6.5.1 Qualitative analysis: An analyte of interest is identified by the comparison of the analytes expected retention time and reference mass spectra.

6.5.1.1 The sample relative retention time (RRT) must compare within  $\pm 0.06$  RRT units of the RRT of the standard component. For reference the daily calibration run RRT is used.

6.5.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% of the base peak for

that component MUST be present in the sample. Also, the relative intensities of ions present in the standard mass spectra must agree to within  $\pm 20\%$  of the sample mass spectra.

6.5.1.3 For samples containing components not associated

with the calibration standards, a library search may be made for the purpose of tentative identification.

#### 6.5.2 Quantitative analysis

6.5.2.1 When a compound has been identified, quantification will take place using the internal standard technique. The internal standard nearest the retention time of the constituent is used.

6.5.2.2 The concentration of each analyte is calculated by the data system using the following:

$$\text{concentration (ug/l)} = \frac{(ax)(is)}{(ais)(rf)(vo)} \times \text{Dilution factor}$$

where:

ax = Area of characteristic ion for compound

is = Amount of internal standard injected

ais = Area of characteristic ion for the internal std.

rf = Response factor for compound

vo = Volume of water purged

### 7.0 Quality control

7.1 Before analysis begins a reagent water blank is run in order to ascertain that interferences from the analytical system, glassware, and reagents are under control (ie. below detection limits). Each time a set of samples is run or there is a change in reagents, a reagent water blank must be run in order to evaluate contamination problems.

7.2 Each day that analysis is performed, the daily midpoint calibration standard is run in order to evaluate the initial calibration curve and to ensure that the GC/MS system is working properly. The analysts judgement and experience is crucial certain judgements must be made by the analyst. Does the chromatography look correct? Is the response obtained comparable to that obtained in the past? Have the constituent retention

times shifted substantially? Is there any background contamination? If any changes to the analytical system are made recalibration of the system must take place.

#### 7.3 Required instrument QC

7.3.1 The mass spectrometer must meet the ion abundance criteria set forth by the EPA.

7.3.2 There must be an initial calibration of the GC/MS system.

7.3.3 On each working day the reagent water blank must be run to verify that no contamination is present.

7.3.4 On each working day the initial calibration must be verified by running a midpoint calibration standard.

7.3.5 For each analytical batch for up to 20 samples per batch a matrix spike and matrix spike duplicate must be run on the matrix being analyzed.

7.4 The analyst on an ongoing process must analyze a reagent blank, a matrix spike, and a matrix spike duplicate for each analytical batch.

7.4.1 The QC acceptance criteria for spike/spike duplicates are taken straight from EPA method 8240.

<u>Compound</u>	<u>Limit for s</u>	<u>Range for x</u>
1,1-dichloroethene	4.6	d-150%
1,2-dichloroethene	2.8	54-156%
1,2-dichloroethane	3.0	49-155%
carbon tetrachloride	2.6	70-140%
1,1,2-trichloroethane	2.7	52-150%
tetrachloroethene	2.5	64-148%
chlorobenzene	3.2	37-160%
ethylbenzene	3.8	37-162%
1,3-dichlorobenzene	2.8	59-156%
1,4-dichlorobenzene	3.6	18-190%

7.5 On an ongoing basis the analyst must analyze a QC check sample at the midrange concentration level at least once every two weeks. The percent recoveries for all analytes must be between 60-140% of the actual value.

7.6 To determine acceptable accuracy and precision limits for

surrogate standards the following procedure is used.

7.6.1 For each sample analyzed, calculate the percent recovery for each surrogate in the sample.

7.6.2 Once a minimum of 30 sample of the same matrix type are analyzed, the average percent recovery is calculated and the standard deviation of the percent recovery for each of the surrogates.

7.6.3 For a given matrix, the upper and lower control limits for method performance for each surrogate standard is calculated as follows:

Upper control limit (UCL) = AVE + 3s

Lower control Limit (LCL) = AVE - 3s

7.6.4 For aqueous and soil matrices, the UCL and LCL calculated from the above are compared with the control limits listed below which should have a wider range and can be found in EPA method 8240:

<u>Surrogate compound</u>	<u>Low/Medium water</u>	<u>Low/Medium soil</u>
4-Bromofluorobenzene	86-115%	74-121%
1,2-dichloroethane (d4)	76-114%	70-121%
Toluene (d8)	88-110%	81-117%

## 8.0 Instrument logs

8.1 Each analyst is expected to keep up to date the following instrument logs. The analysis run logs for each method. The QC log book. The section of the GC/MS maintenance log that pertains to the instrument being used, and the GC/MS standards log book.

8.1.1 The analysis run log book contains all the samples, spikes, blanks, and standards run on the particular instrument involved. This log book also contains the instrument upkeep records (ie. septum changes, injector liner changes, source cleaning, and column replacement)

8.1.2 The QC log book contains the initial calibration

and the continuing calibrations associated with the instrument involved. It also contains any pertinent control charts. There is also an auxiliary QC log book which contains the BFB std. area log, the internal std.'s log, and the surrogate std.'s log. These logs are updated continually as analyses are completed.

8.1.3 The GC/MS maintenance log contains any down time and services performed by any outside service personnel (ie. hardware problems).

8.1.4 The GC/MS standards log book contains the source, date made and related information pertaining to the standards involved in any ongoing analysis. This logbook is maintained in order to be able to locate any problems with standard suppliers std. mixes. (ie. recall of bad batch std.'s)

## SECTION III

## SAMPLE PREPARATION

A. WATER SAMPLE PREPARATION

## 1. Acid Digestion Procedure for Furnace Atomic Absorption Analysis

Shake sample and transfer 100 mL of well-mixed sample to a 250-mL beaker, add 1 mL of (1+1)  $\text{HNO}_3$  and 2 mL 30%  $\text{H}_2\text{O}_2$  to the sample. Cover with watch glass or similar cover and heat on a steam bath or hot plate for 2 hours at  $95^\circ\text{C}$  or until sample volume is reduced to between 25 and 50 mL, making certain sample does not boil. Cool sample and filter to remove insoluble material. (NOTE: In place of filtering, the sample, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.) Adjust sample volume to 100 mL with deionized distilled water. The sample is now ready for analysis.

Concentrations so determined shall be reported as "total."

If Sb is to be determined by furnace AA, use the digestate prepared for ICP/flame AA analysis.

## 2. Acid Digestion Procedure for ICP and Flame AA Analyses

Shake sample and transfer 100 mL of well-mixed sample to a 250-mL beaker, add 2 mL of (1+1)  $\text{HNO}_3$  and 10 mL of (1+1)  $\text{HCl}$  to the sample. Cover with watch glass or similar cover and heat on a steam bath or hot plate for 2 hours at  $95^\circ\text{C}$  or until sample volume is reduced to between 25 and 50 mL, making certain sample does not boil. Cool sample and filter to remove insoluble material. (NOTE: In place of filtering, the sample, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.) Adjust sample volume to 100 mL with deionized distilled water. The sample is now ready for analysis.

Concentrations so determined shall be reported as "total."

B. SOIL/SEDIMENT SAMPLE PREPARATION

## 1. Acid Digestion Procedure for ICP, Flame AA and Furnace AA Analyses

## a. Scope and Application

This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic absorption spectroscopy (AAS) or by inductively coupled plasma spectroscopy (ICP). Samples prepared by this method may be analyzed by AAS or ICP for the following metals:

## CHROMIUM

## Method 218.2 CLP-M\* (Atomic Absorption, Furnace Technique)

Optimum Concentration Range: 5-100 ug/L

Approximate Detection Limit: 1 ug/L

Preparation of Standard Solution

1. Stock solution: Prepare as described under Part C methods, AA Flame Technique.
2. Calcium Nitrate solution: Dissolve 11.8 grams of calcium nitrate,  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  (analytical reagent grade) in deionized distilled water and dilute to 100 mL. 1 mL = 20 mg Ca.
3. Prepare dilutions of the stock chromium solution to be used as calibration standards at the time of analysis. The calibration standards must be prepared using the same type of acid and at the same concentration as will result in the sample to be analyzed after sample preparation. To each 100 mL of standard and sample alike, add 1 mL of 30%  $\text{H}_2\text{O}_2$  and 1 mL of the calcium nitrate solution.

Instrument Parameters (General)

1. Drying Time and Temp: 30 sec @ 125°C.
2. Ashing Time and Temp: 30 sec @ 1000°C.
3. Atomizing Time and Temp: 10 sec @ 2700°C.
4. Purge Gas Atmosphere: Argon
5. Wavelength: 357.9 nm
6. Other operating parameters should be set as specified by the particular instrument manufacturer.

Notes

1. The above concentration values and instrument conditions are for a Perkin Elmer HGA-2100, based on the use of a 20 uL injection, continuous flow purge gas and non-pyrolytic graphite and are to be used as guidelines only.
2. Hydrogen peroxide is added to the acidified solution to convert all chromium to the trivalent state. Calcium is added to a level above 200 mg/L where its suppressive effect becomes constant up to 1000 mg/L.
3. Background correction is required.
4. Nitrogen should not be used as a purge gas because of possible CN band interference.
5. Pipette tips have been reported to be a possible source of contamination.
6. For every sample analyzed, verification is necessary to determine that method of standard addition is not required (see Exhibit E).
7. If method of standard addition is required, follow the procedure given in Exhibit E.

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\*CLP-M modified for the Contract Laboratory Program.

## CHROMIUM

### Method 218.2 (Atomic Absorption, furnace technique)

STORET NO. 01034

Dissolved 01030

Suspended 01031

Optimum Concentration Range: 5–100  $\mu\text{g/l}$

Detection Limit: 1  $\mu\text{g/l}$

#### Preparation of Standard Solution

1. Stock solution: Prepare as described under "direct aspiration method".
2. Calcium Nitrate Solution: Dissolve 11.8 grams of calcium nitrate,  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  (analytical reagent grade) in deionized distilled water and dilute to 100 ml. 1 ml = 20 mg Ca.
3. Prepare dilutions of the stock chromium solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared to contain 0.5% (v/v)  $\text{HNO}_3$ . To each 100 ml of standard and sample alike, add 1 ml of 30%  $\text{H}_2\text{O}_2$  and 1 ml of the calcium nitrate solution.

#### Sample Preservation

1. For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.

#### Sample Preparation

1. Prepare as described under "direct aspiration method". Sample solutions for analysis should contain 0.5% v/v  $\text{HNO}_3$ .

#### Instrument Parameters (General)

1. Drying Time and Temp: 30 sec–125°C.
2. Ashing Time and Temp: 30 sec–1000°C.
3. Atomizing Time and Temp: 10 sec–2700°C.
4. Purge Gas Atmosphere: Argon
5. Wavelength: 357.9 nm
6. Other operating parameters should be set as specified by the particular instrument manufacturer.

#### Analysis Procedure

1. For the analysis procedure and the calculation, see "Furnace Procedure" part 9.3 of the Atomic Absorption Methods section of this manual.

Approved for NPDES and SDWA

Issued 1978

## Notes

1. The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 ul injecton, continuous flow purge gas and non-pyrolytic graphite.
2. Hydrogen peroxide is added to the acidified solution to convert all chromium to the trivalent state. Calcium is added to a level above 200 mg/l where its suppressive effect becomes constant up to 1000 mg/l.
3. Background correction may be required if the sample contains high dissolved solids.
4. Nitrogen should not be used as a purge gas because of possible CN band interference.
5. Pipet tips have been reported to be a possible source of contamination. (See part 5.2.9 of the Atomic Absorption Methods section of this manual.)
6. For every sample matrix analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
7. If method of standard addition is required, follow the procedure given earlier in part 8.5 of the Atomic Absorption Methods section of this manual.
8. For quality control requirements and optional recommendations for use in drinking water analyses, see part 10 of the Atomic Absorption Methods section of this manual.
9. Data to be entered into STORET must be reported as ug/l.

## Precision and Accuracy

1. In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at concentrations of 19, 48, and 77 ug Cr/l, the standard deviations were  $\pm 0.1$ ,  $\pm 0.2$ , and  $\pm 0.8$ , respectively. Recoveries at these levels were 97%, 101%, and 102%, respectively.

## Quality Goals

Method	Analyte	Accuracy MS/MSD %Rec.	Precision RPD	Post Spike Accuracy %Rec.
EPA 218.2*	Chromium	75 - 125	20	85 - 115

PQL - 1.0 µg/L

\* - From CLP SOW

MS - Matrix Spike

MSD - Matrix Spike Duplicate

CCV Recoveries - 80 - 120%

Preparation Blank must less than PQL used

Method	Analyte	Accuracy MS/MSD %Rec.	Precision RPD
EPA 524.2	1,1-Dichloroethene	61 - 145	14
EPA 524.2	Trichloroethene	71 - 120	14
EPA 524.2	Benzene	76 - 117	11
EPA 524.2	Toluene	76 - 125	13
EPA 524.2	Chlorobenzene	75 - 130	13
EPA 524.2	Toluene-d8 (surrogate)	88 - 110	
EPA 524.2	Bromofluorobenzene (surrogate)	86 - 115	
EPA 524.2	1,2-Dichloroethane (surrogate)	76 - 114	

PQL's - 0.5 µg/L for all compounds except  
1,2-dibromo-3-chloropropane - 1.0 µg/L

Preparation Blank analyte values must be less than respective PQL's.

Calibration and tuning protocols will follow CLP SOW.

## Appendix F

### Sampling Results

# PLANT 44-PRIVATE WELL SAMPLING DATA

21-24 MAY 1994

OEMH #	SAMPLING LOCATION/NOTES	VOCs (UG/L)	TTL CR (UG/L)	TRIP BLK	FIELD BLK
GP940150	1st catch-outdoor sample.	1.3-TCE			
GP940151	service house.			ND	
GP940152	Duplicate.	1.2-TCE			
GP940153				ND	
GP940154			11.4		
GP940155	Duplicate.		11.6		
GP940156	VOC field blank				0.55-MECL2
GP940157				ND	
GP940158	Cr field blank				ND (Cr)
GP940159	Sample collected after 10 minute purge.	1.2-TCE			
GP940160				ND	
GP940161	1st catch-Well only used for irrigation.	12-TCE			
GP940162	Owner had been irrigating for three hours			ND	
GP940163	prior to sampling.		1.2		
GP940164	Sample collected after 10 minute purge.	13-TCE			
GP940165				ND	
GP940166	Kitchen tap. Screen removed.	ND			
GP940167				X	
GP940168			1		
GP940169	Outside between well and tank. Sample	ND			
GP940170	collected after pump turned on (5 minutes)			X	
GP940171	No power to pump. 1st catch outside.	31-TCE; 44-CHCL3; 1.2-1,1-DCE; 1.2-DCP; 4.7-MECL2; 44-TTHM			
GP940172				ND	
GP940173			71.5		
GP940174	Sample collected after 15 minute purge.	36-TCE; 55-CHCL3; 1.2-1,1-DCE; 6.4-BENZENE; 55-TTHM			
GP940175				ND	
GP940176	No power to pump. Non-operative since	110-TCE; 7.2-CHCL3; 2.9-1,1-DCE; 7.2-TTHM			
GP940177	Aug 93. Purged 15 min. prior to sample 1.			ND	
GP940178	Duplicate.	120-TCE; 6.3-CHCL3; 2.7-1,1-DCE; 6.3-TTHM			
GP940179				ND	
GP940180			146		
GP940181	Duplicate.		137		
GP940182	Field blank.				1.0-1,1,1-TCA
GP940183				ND	
GP940184	Field blank.				ND (Cr)
GP940185	Collected after additional 10 min. purge.	110-TCE; 7.2-CHCL3; 3.1-1,1-DCE; 7.2-TTHM			

# PLANT 44-PRIVATE WELL SAMPLING DATA

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GP940186	All samples inside per DEQ. AL noted an	ND		ND	
GP940187	outdoor spigot.				
GP940188	Duplicate.	ND		X	
GP940189					
GP940190				X	
GP940191				12	
GP940192	Duplicate.			10.6	
GP940193	Field blank.				ND
GP940194					
GP940195	Field blank.				ND (Cr)
GP940196	Collected after 10 minute purge.	ND			
GP940197				X	
GP940198	Kitchen tap after outdoor filter,	ND			
GP940199	water softener, and carbon filter.			X	
GP940200			ND		
GP940201	Outdoor location-purged tank and filters 1	2.9-TCE			
GP940202	min. before pumped turned on.				ND
GP940203	School breakroom sink.	ND			
GP940204				X	
GP940205				4	
GP940206	Outdoor sample collected after draining	ND			
GP940207	holding tank and pump turning on-10 min.			X	
GP940208	Kitchen tap. Has water softener and	ND			
GP940209	filter under sink.			X	
GP940210			ND		
GP940211	Owner had been irrigating for 2 hours.	ND			
GP940212	Pump cycling continuously-No purge.			X	
GP940213	Outside locations only-used for irrigation.	ND			
GP940214				X	
GP940215				1.2	
GP940216		ND			
GP940217				X	
GP940218	Kitchen tap.	ND			
GP940219				X	
GP940220				2.8	
GP940221	Outside before holding tank.	ND			
GP940222				X	

**PLANT 44-PRIVATE WELL SAMPLING DATA**  
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GP940223	Kitchen tap. Water is chlorinated-no sodium thiosulfate added.	ND			
GP940224				X	
GP940225					
GP940226	Duplicate.			5.4	
GP940227	Field blank.			5.2	
GP940228	Outside before holding tank.	ND			ND (Cr)
GP940229				X	
GP940240	Kitchen tap. Water is chlorinated-no sodium thiosulfate added. Screen removed	ND			
GP940241	Duplicate.				
GP940242				X	
GP940243					
GP940244				3.5	
GP940245	Duplicate.			3.4	
GP940246	Field blank.				ND
GP940247				X	
GP940248	Field blank.				ND (Cr)
GP940249	Outside before holding tank. Waited for pump to cycle-about 31 minutes.	3.6-TCE			
GP940250	Outside-irrigation only.			ND	
GP940251		49-TCE; 5.5-1,1-DCE			
GP940252				ND	
GP940253				9	
GP940254	After 10 minute purge.	49-TCE; 5.2-1,1-DCE			
GP940255				ND	
GP940256	Kitchen tap. Unable to remove screen.	1.7-TCE			
GP940257				ND	
GP940258				4.6	
GP940259	Outside before holding tank. 10 minute purge	1.1-TCE			
GP940260	Kitchen tap. Screen removed.	ND		ND	
GP940261				X	
GP940262					
GP940263				6.4	
GP940264	Outside before holding tank. Ran three hoses to cycle pump. 10 minute purge.	ND			
GP940265	Line to house not functional. Samples collected from tap after holding tank.	ND		X	
GP940266					
GP940267				X	
GP940268				1.2	
GP940269	After 10 minute purge.	ND			

# PLANT 44-PRIVATE WELL SAMPLING DATA

GP940270						X	
GP940271	Collects water outdoors for use indoors.		ND				
GP940272	Pipe from ground, elbow attached.					X	
GP940273					3.5		
GP940274	After 10 minute purge.		ND				
GP940275						X	
GP940276	All samples at kitchen tap. Screen removed.		ND				
GP940277						X	
GP940278	After 10 minute purge.		ND				
GP940279						X	
GP940280					2.7		
GP940281	Duplicate.				2.8		
GP940300	Drinking fountain in day care center.		ND				
GP940301						X	
GP940302	Duplicate.		ND				
GP940303						X	
GP940304					2.1		
GP940305	Duplicate.				11.5		0.53-MECL2
GP940306	Field blank.						
GP940307						ND	
GP940308	Field blank.						ND (Cr)
GP940309	10 minute purge to allow pump to cycle.		ND				
GP940310	Rain delay between replicates.					X	
GP940311	Kitchen tap-no screen.		ND				
GP940312						X	
GP940313					ND		
GP940314	Outside before holding tanks.		ND				
GP940315						X	
GP940316	All samples indoors at breakroom sink.		ND				
GP940317	Screen removed.					X	
GP940318	Duplicate.		ND				
GP940319						X	
GP940320					1		
GP940321					1		
GP940322	After 10 minute purge.		ND				
GP940323						X	

# PLANT 44-PRIVATE WELL SAMPLING DATA

ND--None Detected			
X--Not Analyzed--Trip blanks not analyzed if accompanying samples are ND			
TTHM--Total Trihalomethanes			